WEST

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Search Results - Record(s) 1 through 1 of 1 returned.

	m43.	I.O.DE		Feb 23, 199
L23: Entry 1 of 1	File: V	JSPI		100 20, 100
US-PAT-NO: 5874106 DOCUMENT-IDENTIFIER: US 5874106	A			
TITLE: Filled gelatin capsules				
DATE-ISSUED: February 23, 1999				
INVENTOR-INFORMATION:				
NAME	CITY	STATE	ZIP CODE	COUNTRY
Adesunloye; Adedotun Tony	Aurora	CO		
Stach; Paul Edward	Broomfield	CO		
US-CL-CURRENT: 424/456; 424/408	514/962			

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Term	Documents
BENZOIC.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	64709
BENZOICS.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	7
ACID[USPT,PGPB]	552538
ACIDS[USPT,PGPB]	313052
(10 AND (BENZOIC ADJ (ACID[CLM]))) USPT,PGPB,JPAB,EPAB,DWPI,TDBD.	I

Display 10 Documents, starting with Document: 1

Display Format: Change Format

HVI : Hall mA

Taybitz does not mentioned the (claimed) particle size of metallic particles and mineral metallic particles and mineral metallic particles and metallic particles and metallic particles and mineral metallic particles and metallic particles.

Levine discloses that a preferred metallic pigment should have a diameter of 10-20

microns (see Levine, col. 5, lines 30-34).

Kohara discloses that the average diameter of a fibrous filler is 0.1 to 100 micrometers, and the length is from 5 micrometers to 20 millimeters. Average article diameter of powdery

filler is 0.01 micrometer to 1 millimeter (see Kohara, col. 4, lines 27-38).

Clark discloses thermoplastic compositions having a speckled surface appearance; This appearance results torm the use of nondispersing pigments (abstract; col. 10, lines 1-2). It is preferred that these pigments possess a large aspect ratio, greater than about 20 (col. 10, lines 33-preferred that these pigments possess a large aspect ratio, greater than about 20 (col. 10, lines 33-preferred that these pigments possess a large aspect ratio, greater than about 20 (col. 10, lines 33-preferred that these pigments possess a large aspect ratio, greater than about 20 (col. 10, lines 33-preferred that these pigments possess a large aspect ratio, greater than about 20 (col. 10, lines 33-preferred that these pigments possess a large aspect ratio, greater than about 20 (col. 10, lines 33-preferred that these pigments possess a large aspect ratio, greater than about 20 (col. 10, lines 33-preferred that these pigments possess a large aspect ratio, greater than a preferred that these pigments possess a large aspect ratio, greater than a preferred than a preferred than a preferred that the preferred than a pr

.(8£

Based on the teachings of Levine, Kohara and Clark, it is the examiner's position that it would have been obvious to one ordinary skill in the art to use in the composition of Taubitz metallic particles of claimed particle size, fibers of claimed average diameter and average aspect ratio and interals particles of claimed average particle size in order to produce products having bright reflective appearance, enhanced hiding power; improved crack resistance and moldability as well as better speckled appearance.

4. Any inquiry concerning this communication or earlier communications from the examiner should be directed to U.K. Rajguru whose telephone number is (703) 308-3224. The examiner can normally be reached on Monday - Friday from 9:30 am to 6:00 pm.

WEST

Generate Collection

Search Results - Record(s) 1 through 10 of 11 returned

1. Document ID: US 6211268 B1

L17: Entry 1 of 11

File: USPT

Apr 3, 2001

US-PAT-NO: 6211268

DOCUMENT-IDENTIFIER: US 6211268 B1

TITLE: Polyoxymethylene resin composition

DATE-ISSUED: April 3, 2001

INVENTOR-INFORMATION:

COUNTRY STATE ZIP CODE CITY NAME JPX Matsumura; Takatoshi Yokkaichi JPX Nishizawa; Chiharu Yokkaichi JPX Mimura; Hiroshi Yokkaichi JPX Yokkaichi Yada; Hiroshi JPX Kurashige; Kazuo Yokkaichi

US-CL-CURRENT: 524/100; 524/275, 524/377, 524/378, 524/384, 524/394, 524/400

Full Title Citation Front Review Classification Date Reference Claims KMC Draw Desc Image

2. Document ID: US 6077908 A

L17: Entry 2 of 11

File: USPT

Jun 20, 2000

US-PAT-NO: 6077908

DOCUMENT-IDENTIFIER: US 6077908 A

TITLE: Polyoxymethylene resin composition

DATE-ISSUED: June 20, 2000

INVENTOR-INFORMATION:

NAME

CITY

STATE ZIP CODE

COUNTRY

Yahiro; Shyuzi

Kurashiki

JPX

US-CL-CURRENT: 525/218; 525/282, 528/310, 528/322

Full Title Citation Front Review Classification Date Reference Claims MMC Draw Desc Image

3. Document ID: US 5310823 A

L17: Entry 3 of 11

File: USPT

May 10, 1994

can be found in a prior Office action.

A request for reconsideration has been filed on May 23, 2000 (paper no. 4). Ι.

The text of those sections of Title 35, U.S. Code not included in this action .2

Claims 1-12 are rejected under 35 U.S.C. 103(a) as being unpatentable over

This rejection is incorporated hereby reference from prior office action paper Morris (USP 3919143) in view of EP 811659 and Yogishita et al (USP 4931509).

.£ .on

polyvalent carboxylic acid is used. persuasive when Morris does specifically state in col. 5, lines 12-145 that on carboxylic acid salts and not acids as required by instant claim 1. This is not Applicants state that (page 3, paragraphs 1-3) Morris uses polyvalent

Therefore applicants' other argument that "Morris presents only one example as an

Examples in prior art are considered illustrative and not limitative.

acid" is not persuasive.

hydrotalcite does not improve compression set. This comment is true but relevant Applicants comment (p. 4, paragraph 5), that EP '659 discloses that

US-PAT-NO: 5310823

DOCUMENT-IDENTIFIER: US 5310823 A

TITLE: Polyacetal resin composition

DATE-ISSUED: May 10, 1994

INVENTOR-INFORMATION:

NAME

Kunitomi; Masaki

Ishii; Hiromitsu

Yamamoto; Yoshiyuki

CITY

STATE

ZIP CODE

JPX

JPX

JPX

JPX

US-CL-CURRENT: $\underline{525}/\underline{400}$; $\underline{524}/\underline{155}$, $\underline{524}/\underline{157}$, $\underline{524}/\underline{158}$, $\underline{524}/\underline{161}$, $\underline{524}/\underline{282}$, $\underline{525}/\underline{80}$,

<u>525/90, 525/93</u>



File: USPT

4. Document ID: US 4987174 A

L17: Entry 4 of 11

US-PAT-NO: 4987174 DOCUMENT-IDENTIFIER: US 4987174 A

TITLE: Low gloss polyacetal resin

DATE-ISSUED: January 22, 1991

INVENTOR-INFORMATION:

NAME CITY STATE ZIP CODE COUNTRY

Ying; Edwina Bridgewater NJ Hayes; Conrad G. Plainfield NJ

US-CL-CURRENT: 524/444; 524/450, 524/593



5. Document ID: US 4855365 A

L17: Entry 5 of 11

File: USPT

Aug 8, 1989

Jan 22, 1991

IIVI : tinU thA

will the statutory period for reply expire later than SIX MONTHS from the mailing date of this final action.

4. Any inquiry concerning this communication or earlier communications from

4. Any inquiry concerning this communication or earlier communications from the examiner should be directed to U.K. Rajguru whose telephone number is (703) 308-3224. The examiner can normally be reached on Monday - Friday from 9:30 am to 6:00 pm.

If attempts to reach the examiner by telephone are unsuccessful, the

examiner's supervisor, Jim Seidleck, can be reached on (703) 308-2462. The fax phone number for the organization where this application or proceeding is assigned is (703) 305-3599.

proceeding should be directed to the receptionist whose telephone number is (703)

Any inquiry of a general nature or relating to the status of this application or

1990-808

U.K. Rajguru/om August 18, 2000 US-PAT-NO: 4855365

DOCUMENT-IDENTIFIER: US 4855365 A

TITLE: Poly-.beta.-alanine compound, a process for producing the same and a polyacetal resin composition containing a poly-.beta.-alanine compound

DATE-ISSUED: August 8, 1989

INVENTOR-INFORMATION:

NAME

Kawasaki

CITY

ZIP CODE STATE

COUNTRY

Yamamoto; Fumihiko

JPX

Misumi; Teruyuki

Yokohama

JPX

 $\text{US-CL-CURRENT: } \underline{525/401}; \ \underline{525/393}, \ \underline{525/402}, \ \underline{525/427}, \ \underline{525/472}, \ \underline{526/192}, \ \underline{526/210},$ 526/212, 528/310, <u>528/328</u>, <u>528/332</u>



6. Document ID: JP 2000169667 A

L17: Entry 6 of 11

File: JPAB

Jun 20, 2060

PUB-NO: JP02000169667A

DOCUMENT-IDENTIFIER: JP 2000169667 A

TITLE: REINFORCED POLYACETAL RESIN COMPOSITION AND INJECTION MOLDED PRODUCT

PUBN-DATE: June 20, 2000

INVENTOR-INFORMATION:

NAME

COUNTRY

YAMANAKA, TORU MAKABE, YOSHIKI

NISHIMURA, TORU

INT-CL (IPC): C08L 59/00; B29C 45/00; C08K 3/02; C08K 3/34; C08K 5/13; C08K 5/17; C08K 5/3492; C08K 5/521





Document ID: JP 11100 486 A

L17: Entry 7 of 11

File: DWPI

Apr 13, 1999

) Heast etain the 20 projest

Application/Control Number: 09/066275

IIVI : iiuU nA

1. A request for extension of time (paper no. 8) and an amendment (paper no. 9) have been

filed on May 11, 2000.

Applicant's election with traverse of claims 1-9 are in Paper No. 9 is acknowledged. The traversal is on the ground(s) that it would not constitute an undue burden to search all the subject

matter as claimed. This is not found persuasive because the applicants have made only a conclusionary statement without any support for it. There is actually an undue burden for search

for non-elected claim 10.

The requirement is still deemed proper and is therefore made FINAL.

Rejection of claim under 35 U.S.C. 112, second paragraph (see section 3, page 3 of office

action paper no. 5) is now withdrawn.

4. The text of those sections of Title 35, U.S. Code not included in this action can be found

in a prior Office action.

Claims 1-9 and (newly added) 11-14 are rejected under 35 U.S.C. 103(a) as being

unpatentable over Schlumpf (Canadian Patent 2016447) in view of Shaw et al (USP 4643940),

This rejection (also to be applied to new claims 11-14) is incorporated here by reference

M from section 4, pages 3-5, of prior office, paper no. 5

Hosoda et al (USP 3812225) and Figjii et al (USP 4680318).

Applicant's arguments filed May 11, 2000 (paper no. 9) have been fully considered but

they are not persuasive.

DERWENT-ACC-NO: 1999-296594

DERWENT-WEEK: 199928

COPYRIGHT 2001 DERWENT INFORMATION LTD

TITLE: Polyoxymethylene resin composition - with good heat stability and

moldability

PRIORITY-DATA: 1997JP-0263431 (September 29, 1997)

PATENT-FAMILY:

PUB-NO

PUB-DATE

LANGUAGE

PAGES MAIN-IPC

JP 11100486 A April 13, 1999

008 C08L059/00

INT-CL (IPC): C08K 5/15; C08K 5/29; C08L 59/00



KAMIC - Drawn Desc - Image |

8. Document ID: JP 07188514 A

L17: Entry 8 of 11

File: DWPI

Jul 25, 1995

DERWENT-ACC-NO: 1995-290564

DERWENT-WEEK: 199538

COPYRIGHT 2001 DERWENT INFORMATION LTD

TITLE: Polyacetal resin compsn. used for moulding car electrical parts comprising hindered phenol type antioxidant and formaldehyde scavenger

PRIORITY-DATA: 1993JP-0333738 (December 27, 1993)

PATENT-FAMILY:

PUB-NO

PUB-DATE

LANGUAGE

PAGES MAIN-IPC

JP 07188514 A

July 25, 1995

008

C08L059/00

INT-CL (IPC): C08K 5/13; C08L 59/00





9. Document ID: JP 06256621 A

L17: Entry 9 of 11

File: DWPI

Sep 13, 1994

HVI : JinU nA

Applicant's arguments do not comply with 37 CFR 1.111(c) because they do not clearly point out the patentable novelty which he or she thinks the claims present in view of the state of the art disclosed by the references cited or the objections made. Further, they do not show how the amendments avoid such references or objections.

On page 6, paragraph 2 of above response paper no. 9, the applicants argue that Schlumpf

teaches away from claimed invention because Schlumpf discloses solids at lease 92.1%. While there is a difference of only 0.1% (between claimed 92.0% and disclosed 92.1%), this difference fails to distinguish patentably the instant invention from the prior art, particularly because the applicants have not established the criticality of the upper limit viz. 92.0%.

Applicants' further argue in the next paragraph on the same page, that Schlumpf does not continue that argue in the next paragraph on the same page, that Schlumpf does not continue that argue in the next paragraph on the same page, that Schlumpf does not continue that argue in the next paragraph on the same page, that Schlumpf does not continue that argue in the next paragraph on the same page, that Schlumpf does not continue that argue in the next paragraph on the same page, that Schlumpf does not continue that argue in the next paragraph on the same page, that Schlumpf does not continue that argue in the next paragraph on the same page.

discloses a calcium carbonate of a mean statistical particle diameter of 0.5 - 50 microns. This range overlaps that of calcium carbonate (viz. 0.3 to 18 microns) in example 1 in instant specification page 14. Furthermore also the applicants have not shown any criticality of having

the only the claimed particle size distribution.

On page 6, paragraph 4 of same paper, the applicants state that apparatus of Schlumpf

cannot degas the composition and therefore Schlumpf teaches away form infinelusion of a blowing agent. Wether the apparatus can or cannot degas the composition has no influence at all.

DERWENT-ACC-NO: 1994-330256

DERWENT-WEEK: 199441

COPYRIGHT 2001 DERWENT INFORMATION LTD

TITLE: Polyoxymethylene resin compsn. for moulding e.g. car parts - includes a hypophosphite cpd., hindered phenol and amine cpds. and a formaldehyde scavenger

PRIORITY-DATA: 1993JP-0043688 (March 4, 1993)

PATENT-FAMILY:

 PUB-NO
 PUB-DATE
 LANGUAGE
 PAGES
 MAIN-IPC

 JP 06256621 A
 September 13, 1994
 008
 C08L059/00

INT-CL (IPC): C08K 5/52; C08L 59/00





10. Document ID: JP 3168639 B2, JP 05125255 A

L17: Entry 10 of 11

File: DWPI

May 21, 2001

DERWENT-ACC-NO: 1993-200634

DERWENT-WEEK: 200130

COPYRIGHT 2001 DERWENT INFORMATION LTD

TITLE: Polyoxymethylene resin compsn. for reduced formaldehyde odour and mould deposits - comprises polyoxymethylene resin contg. oxazoline and/or oxadiazoline cpd. hindered phenol and amine cpds. and formaldehyde scavenger for heat resistance

PRIORITY-DATA: 1991JP-0291568 (November 7, 1991)

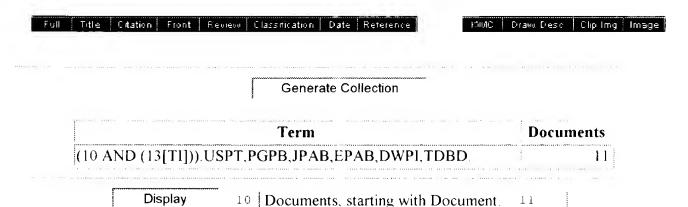
PATENT-FAMILY:

 PUB-NO
 PUB-DATE
 LANGUAGE
 PAGES
 MAIN-IPC

 JP 3168639 B2
 May 21, 2001
 010
 C08L059/00

 JP 05125255 A
 May 21, 1993
 010
 C08L059/00

INT-CL (IPC): C08K 5/13; C08K 5/18; C08K 5/35; C08L 59/00



ITTI :tinU nA

Any inquiry of a general nature or relating to the status of this application or proceeding

should be directed to the receptionist whose telephone number is (703) 308-0661.

U.K. Rajguru/om August 14, 2000

DB Name	Query	<u>Hit</u> Count	<u>Set</u> <u>Name</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	(polyacetal resin or polyoxymethylene resin) and (anthranilic acid or 2 adj amino benzoic acid or 4 adj amino benzoic acid)	21	<u>L19</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	(polyacetal resin or polyoxymethylene resin) and (anthranilic acid or 4 adj amino benzoic acid) near15 low formaldehyde	0	<u>L</u> 18
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	(anthranilic acid or 4 adj amino benzoic acid) near 15 low formaldehyde	0	L <u>17</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	(anthranilic acid or 2 adj amino benzoic acid) near15 low formaldehyde	0	<u>L</u> 16
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	(anthranilic acid or 2 adj amino benzoic acid) near5 low formaldehyde	0	<u>L1</u> 5
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	(anthranilic acid or 2 adj amino benzoic acid) and low formaldehyde	8	<u>L14</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	11 and low formaldehyde	7	<u>L13</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	11 and composit\$3 and article and (mould?? or mold??)and low formaldehyde	0	<u>L12</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	11 and composit\$3 and article and (mould?? or mold??)	37	LII
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	11 and composit\$3 and article	62	<u>L10</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	11 and composit\$3	164	<u>L9</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	polyacetal and 4 adj amino benzoic acid [clm]	0	<u>L8</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	polyacetal and 4 adj amino benzoic acid [ab]	1	<u>L7</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	polyacetal and 4 adj amino benzoic acid [ti]	0	<u>L6</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	polyacetal and 4 adj amino benzoic acid	21	<u>L5</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	polyacetal and (anthranilic acid or 4 adj amino benzoic acid) [clm]	1	L <u>4</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	polyacetal and (anthranilic acid or 4 adj amino benzoic acid) [ab]	3	<u>L3</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	polyacetal and (anthranilic acid or 4 adj amino benzoic acid) [ti]	1	<u>L2</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	polyacetal and (anthranilic acid or 4 adj amino benzoic acid)	260	IJ

examination purposes as indicated is proper.

proper.

variants. Should applicant traverse on the ground that the species are not patentably distinct, applicant should submit evidence or identify such evidence now of record showing the species to be obvious variants or clearly admit on the record that this is the case. In either instance, if the examiner finds one of the inventions anticipated by the prior art, the evidence or admission may be used in a rejection under 35 U.S.C. 103(a) of the other invention.

Because these inventions are distinct for the reasons given above and have acquired a

separate status in the art because of their recognized divergent subject matter, restriction for

Because these inventions are distinct for the reasons given above and the search required for Group I or II is not required for Group II, restriction for examination purposes as indicated is

A telephone call was made to Attorney Arnold Turk on August 9, 2000 to request an oral election to the above restriction requirement, but did not result in an election being made.

Any inquiry concerning this communication or earlier communications from the

examiner should be directed to U.K. Rajguru whose telephone number is (703) 308-3224. The examiner can normally be reached on Monday - Friday from 9:30 am to 6:00 pm. If attempts to reach the examiner by telephone are unsuccessful, the examiner's

supervisor, Jim Seidleck, can be reached on (703) 308-2462. The fax phone number for the organization where this application or proceeding is assigned is (703) 305-3599.

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End of Result Set

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L15: Entry 2 of 2

File: DWPI

09/852383

DERWENT-ACC-NO: 1968-12294Q

DERWENT-WEEK: 196800

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TITLE: Polyoxymethylene stabilisation with aliphatic or

PATENT-ASSIGNEE:

ASSIGNEE

CODE

MTTR

MITR

MITSUBISHI RAYON CO LTD

MITR

PRIORITY-DATA: 1964JP-0025050 (May 4, 1964)

PATENT-FAMILY:

PUB-NO

PUB-DATE

LANGUAGE

PAGES

MAIN-IPC

JP 68020308 B

ABSTRACTED-PUB-NO: JP68020308B

BASIC-ABSTRACT:

Polyoxymethylene of high mol. wt. having at least 1 terminal OH gp. is stabilised with aliphatic or aromatic dicarboxylic acid imide or o-sulphobenzimide, opt. ring substd., using 3-20% ags. NH4OH as reaction medium.

Pref. 10 parts polyoxymethylene, 10-1000 parts 3-20% NH4OH and 3-0.1 parts stabiliser heated and stirred at 50-100 deg.C, esp. 80-90 deg.C for 0.5-5 hr. in sealed tube, opt. under N2. Suitable stabilisers include phthalimide and 3-chloro-phthalimide, malonimide, succinimide, glutamimide, adipimide and o-sulphobenzimide opt. ring substd. by alkyl, alkenyl, cycloalkyl, allyl, amino, acylamine or COOH. Polyoxymethyl ene is pref. reduced to particle size below 50 mesh.

TITLE-TERMS : POLYOXYMETHYLENE STABILISED ALIPHATIC

DERWENT-CLASS: A00

CPI-CODES: A05-H02; A08-A01; A09-A01; A10-E01;

Multipunch Codes: 01- 080 138 180 231 250 273 31- 329 331 359 393 541 546 681 688

720 721

preferably alkylene. urethanylene, ureylene, and combinations thereof. E is nyl, sulfoxy, imino, sulfonsmido, scylimino, scyloxy, ene, arylene, aralkylene, oxy, oxo, hydroxy, thio, sulfo-E include straight chain, branched chain or cyclic alkylcal oligomerization. Examples of structure suitable for

20 greater than 1. 97-100 below), the number of Z groups is preferably valent polymeric chain (e.g., as set forth in Examples an instance wherein the group R is a mono, di, or polyconnection with Schemes I and II below. However, in These embodiments are discussed in detail below in II, or through group X in a compound of Formula III. tion through group E in a compound of Formulas I or bly being bonded to the fluorochemical oligomer porferred that there be I group Z in a compound, prefera-Suitable groups a include non-reactive groups such as 10 Rg, Rg, Q, X and/or E. In most embodiments, it is prefluorochemical oligomeric portion through any of R_L, contain a plurality of Z groups each bonded to the As illustrated in Formulas I-III, a compound can

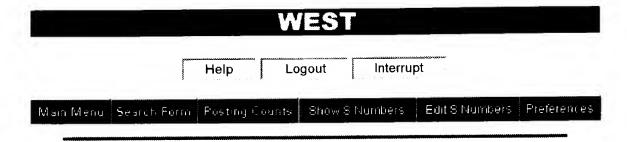
various steps. steps. The schemes show only the major products of the shown in the schemes will be produced by the various Linking group E preferably contains from 1 to about 30 such a functional group. Products other than those capable of being converted or further elaborated into tional group capable of reacting with an isocyanate or the proviso that at least one thereof possesses a funcleast one thereof has an unsatisfied valence, and with Q, R3, X, and E, respectively, absent the proviso that at Rs. Rs G, Rr. Y and L generally correspond to R1, R2. R. Q. R., R. X. E. R. and R are as defined above, and shown in Schemes I and II below, wherein n, a, b, q, R_I, Compositions of the invention can be prepared as

> designates an initiating radical formed upon homolytic the term "group derived from a free-radical initiator" vide initiating radicals upon homolysis. As used herein, peroxyesters, dialkyl peroxides) and the like that procompounds, organic peroxides (e.g., diacyl peroxides, any of the conventional compounds such as organic azo used herein, the term "free-radical initiator" designates X is a group derived from a free-radical initiator. As

[1,1-bis(hydroxymethyl)ethyl]propionamide). yethyl]propionamide]; and 2,2'-azobis{2-methyl-Nazobis {2-methyl-W-[1, 1-bis(hydroxymethyl)-2-hydrox-2,2'-azobis[2-(hydroxymethyl)propionitrile]; exobis[2-methyl-N-(2-hydroxyethyl)propionamide]; yethyl)-2-methylpropionamidine]dihydrochloride; 2,2'pionamidine]dihydrochloride; 2,2'-azobis[N-(2-hydrox-2,2'-azobis[2-methyl-N-2-propenylpro-(+-aminophenyl)-2-methylpropionamidine]tetrahydromethylpropionamidine]dihydrochloride; 2,2'-azobis[Ndihydrochloride; 2,2'-azobis[N-(4-hydroxyphenyl)-2azobis[N-(4-chlorophenyl)-2-methylpropionamidine]other known functional are compounds such as 2,2'rived from axoisobutyronitrile), and those derived from 15 from azo-4-cyanoisovaleric acid), —C(CH₃)₂ CN (degroups such as —C(CH3)(CN)CH2CH2CO2H (derived zoyloxy (derived from benzoyl peroxide), and reactive t-butoxy (derived from di-t-butylpetoxide), and bendecomposition of a free-radical initiator.

tional groups that substantially interfere with free-radicombination thereof, and E is preferably free of funcnitrogen-, sulfur-, or silicon-containing groups or a 20 carbon atoms. E can optionally contain oxygen-,

ferred groups X include those enumerated above.



Search Results -

Term	Documents
ODOR.DWP1,TDBD,EPAB,JPAB,USPT,PGPB	46240
ODOUR.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	20320
ODOURS.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	5195
ODORS.DWPI,TDBD,EPAB,JPAB,USPT,PGPB	15071
REDUCING DWPI, TDBD, EPAB, JPAB, USPT, PGPB.	1161873
REDUCINGS DWPI, TDBD, EPAB, JPAB, USPT, PGPB.	10
(14 AND (ODOR ADJ REDUCING)).USPT,PGPB,JPAB,EPAB,DWPI,TDBD	1

	US Patents Full-Text Database
	US Pre-Grant Publication Full-Text Database
	US Pre-Glant Publication Fills ext Database
	JPO Abstracts Delabase
	EPO Abstracts Database
	Derwent World Patents Index
atabase:	IBM Technical Disclosure Bulletins
it titibuse i	

Da

	114 and odor	reducing		
Refine Search:			 F	Clear

Search History

Today's Date: 12/19/2001

DB Name	Query	Hit Count	Set Name
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	114 and odor reducing	1	<u>L24</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	114 and composit\$3 and molded article	8	<u>L2</u> 3
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	114 and composit\$3	205	<u>L22</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	succinimide same (polyacetal or polyoxymethylene)	3	1.21

Denumuoo-

SCHEME I

Totaitin

oinaga 10

propyleneoxide dihydroacrylate. fluorocyclohexyl acrylate, and tetrameric bexaffuoro-20 CaF175O2NHCH2CH=CH2, and others such as perfluorooctanesulfonamidoethyl vinyl ether, alcohol, perfluorooctyl acrylate, M-methyl perlate and M-methylperfluorooctanesulfonamidoethyl

generally commercially available. Examples of such Compounds of Formula VI are also well known and

oxide and propylene oxide endeapped by hydroxy CARBOWAX TM), block copolymers of ethylene ylene glycols of various molecular weights (available as M-methyl perfluoroccanesulfonamidocthyl methacry- 65 acrylates of methoxypolyethylene glycols and polyethglycol acrylate, mono, di, and polyacrylates and methsuch as ethylene glycol dimethacrylate, triethylene or di (meth) serylates of glycols or polyalkylene glycols methacrylate; acrylamides and methacrylamides; mono 60 methacrylate, trimethylopropane triacrylate, allyl bisphenol A dimethacrylate, isobornyl acrylate and methacrylate, dicyclopentenyl acrylate, ethoxylated dro-s-triazine; special acrylates such as butanediol disuch as isopropyl cyanoacrylate; (tris)-acryloyl-bexahyglutaronitrile, vinylidene cyanide, alkyl cyanoacrylates 2-chloroacrylonitrile, 2-cyanocthylacrylate, methylene ane; nitriles such as acrylonitrile, methacrylonitrile, thoxystlane, and methacryloyloxypropyl trimethoxysilvinyltrichlorosilane, vinyltrimethoxysilane, vinyltrieof Formula VI. In Scheme II, the monomers are the 50 ryl acrylates and methacrylates; olefinic alanes such as hexyl, 2-ethylbexyl, chlorobexyl, octyl, lauryl, or steaate, and vinyl, allyl, methyl, ethyl, butyl, isobutyl, ethyl crotonate, acid methyl maleate, acid butyl itaconhydrides and esters thereof such as dimethyl maleate, 45 maleic, furnarie, itaconie, and citraconic acids, and anacrylic, a-chloro acrylic, a-fluoro acrylic, crotonic, such as vinyl methyl ketone; unsaturated acids such as ethyl vinyl ether, tetrallyloxyethane; vinyl alkyl ketones ether, isobutyl vinyl ether, ethyl vinyl ether, 2-chloro-40 lallyl ethers such as cetyl vinyl ether, dodecyl vinyl as ally acctate and ally heptanoate; alkylvinyl or alkynate, vinyl stearate, divinylearbonate, allyl esters such acetate, vinyl propionate, vinyl isobutyrate, vinyl succinylbenene, M-vinylearbazole; vinyl esters such as vinyl 35 nene, a-methylstyrene, a-cyanomethylstyrene, divilyl chloride; styrene and its derivatives such as vinyltolnylidene fluoride, allyl bromide, allyl chloride, methallides such as vinyl or vinylidene chloride, vinyl or vidimethyl-1,5-hexadiene; vinyl, allyl or vinylidene ha-30 chlorobutadienes, fluoro and difluorobutadienes, 2,5isobutene, butadiene, isoprene, chloro and diency se ethylene, propylene, isobutene, 3-chloro-2lower olefinic hydrocarbons, optionally halogenated, pounds capable of free-radical polymerization, such as 25 compounds include general classes of ethylenic com-

late, the reaction product of isocyanatoethyl methacrylate, M-methylperfluorohexylsulfonamidoethyl acrylate, M-ethyl perfluorooctanesulfonamidoethyl acry-N-methyl perfluorooctanesulfonamidoethyl acrylate, Preferred compounds of Formula v include such as pounds containing fluorinated sulfonsmide groups, ac-Pat. Nos. 2,803,615 (Ahlbrecht et al.) and 2,841,573 55 Compounds of Formula V and methods for the prep-

L(SH)_m, and optionally a non-fluorinated comonomer radical initiator, an end-capping agent of the formula Formula V is oligomerized in the presence of a free-In step (1) of Scheme I, a fluorochemical monomer of

telomer alcohols, fluorochemical thiols, and the like. rylates or methacrylates derived from fluorochemical acrylates, methacrylates, vinyl ethers, and allyl comclude general classes of fluorochemical olefins such as berein by reference. Examples of such compounds in-(Ahlbrecht et al.) which disclosures are incorporated aration thereof are known and disclosed, e.g., in U.S. the initiator is preferably a functional initiator. same, the end-capping agent is optionally present, and

USPT,PGPB,JPAB,EPAB,DWPI,TDBD	succinimide near 15 (polyacetal or polyoxymethylene)	0	<u>L20</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	succinimide near5 (polyacetal or polyoxymethylene)	0	<u>L19</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	114 and formaldehyde scavenger	0	<u>L 18</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	succinimide and (polyacetal or polyoxymethylene) [clm]	14	<u>L17</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	succinimide and (polyacetal or polyoxymethylene) [ab]	4	<u>L</u>]6
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	succinimide and (polyacetal or polyoxymethylene) [ti]	2	<u>L15</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	succinimide and (polyacetal or polyoxymethylene)	230	<u>L14</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	succinimide near formaldehyde scavenger	0	<u>L13</u>
USPT,PGPB	succinimide and formaldehyde scavenger and polyoxymethylene	0	<u>L12</u>
USPT,PGPB	succinimide and formaldehyde scavenger and polyacetal	0	<u>L11</u>
USPT,PGPB	succinimide and formaldehyde scavenger [clm]	0	<u>L10</u>
USPT,PGPB	succinimide and formaldehyde scavenger [ab]	0	<u>L9</u>
USPT,PGPB	succinimide and formaldehyde scavenger [ti]	0	<u>L8</u>
USPT,PGPB	succinimide near13 formaldehyde scavenger	0	<u>L7</u>
USPT,PGPB	succinimide near3 formaldehyde scavenger	0	<u>L6</u>
USPT,PGPB	succinimide near formaldehyde scavenger	0	<u>L5</u>
USPT,PGPB	succinimide and formaldehyde scavenger	11	<u>L4</u>
USPT,PGPB	succinimide and 3406223 [pn]	0	<u>L3</u>
USPT,PGPB	succinimide and 5603927 [pn]	0	<u>L2</u>
USPT,PGPB	succinimide and 5866671 [pn]	0	<u>L1</u>

amino-3-mercaptopropionic acid, and compounds such 2-chloroethanethiol, topropyltrimethoxysilane, captosuccinic acid, 2,3-dimercaptopropanol, 3-mercapmine, 1-chloro-6-mercapto-4-oxahexan-2-ol, 2,3-dimerscid, 12-mercaptododecanoic acid, 2-mercaptoethyladecanol, mercaptoacetic acid, 3-mercaptopropionic propanediol, 11-mercaptoun-←mercaptobutanol, 3-mercapto-1,2-2-mercaptoethanol, Spuisui ethylester. Preferred functionalized end-capping agents ethanedithiol, cysteine, cystein hydrochloride, cysteine ethylcyclohexanedithiol, p-menthane-2,9-dithiol, 1,2thiol, o., m., and p-thiocresol, 2-mercaptoethylamine, acid, 2,5-dimercapto-1,3,4-thiadiazole, 3,4-toluenediacid, 2,3-dimercaptopropanol, 2,3-dimercaptosuccinic N-oxide, 2-mercaptopyridinol, 2-mercaptopyrimidine, thoxysilane, 2-mercaptopyridine, 2-mercaptopyridine-3-mercaptopropyltrimemercaptopropionyl)glycine, mercapto-2-propanol, 2-mercaptopropionic acid, N-(2-8-mercaptomenthone, 2-mercaptonicotinic acid, 4ether, 2-mercaptoethylsulfide, 2-mercaptoimidazole, butanol, 2-mercaptosulfonic scid, 2-mercaptoethyl 3-mercapto-2-2-mercaptobenzoxazole, thiszole,

t-dodecylthiol, 2-mercaptoethyl ether, 2-mercappropanethiol, butanethiol, hexanethiol, n-octylthiol, include mono, di, and polythiols such as ethanethiol, las III or IV) as described below. Such compounds mer) or through the group X in a compound of Formuthrough the fluorochemical or non-fluorinated monogroup that can react with an isocyanate is incorporated bing agents are particularly useful when a functional 30 capable of reacting with an isocyanate. Such end-capresction (e.g., a thiol) but no further functional groups contain a group capable of terminating a radical chain Non-functionalized end-capping agents are those that

about 0.05 to about 0.5 equivalents, preferably about end-capping agent is generally used in an amount of of polymerized monomer units in the oligomer. The to bresent in an amount sufficient to control the number Whether functionalized or not, an end-capping agent toimidazole, and the like.

peroxide and lauroyl peroxide. butylperoxy pathalate, diacylperoxides such as benzoyl ide, peroxyesters such as t-butyl perbenzoate and di-tdialkyl peroxides such as di-t-butyl and dicumyl peroxsuch as cumene, t-butyl, and t-amyl hydroperoxide, lyl maleate; vinyltricthoxyzilane; vinyltrichlotosilane; 50 azo-2-cyanovaleric acid and the like, hydroperoxides fates, azo compounds such as azoisobutyronitrile and are known to those skilled in the art and include persuldefined above in connection with X. Such compounds Also present in step (1) is a free-radical initiator as scid 45 0.25 equivalents, per equivalent of olefinic monomer.

functional), VA-500 (aniline-functional), VR-558 (al-Chemical Industries (Osaka, Japan) as VA-548 (phenol-Some such initiators are available from Wako Pure above in connection with functionalized groups X. able functionalized initiators include those described described thiol end-capping agents. Examples of suittherefore be carried out in the absence of the aboveof a functionalized initiator. Step (1) of Scheme II can be functionalized by way of Scheme II through the use for used. Therefore, a compound of the invention can ing degrees depending on the type and amount of initiaincorporated into the fluorochemical oligomer to vary-The initiating radical formed by an initiator can be

incorporated herein by reference. 4,728,571 (Clemens et al.), the disclosure of which is 15 Corning); and others described in U.S. Pat. No. (Dow Corning); DC-536 siloxane polyamine (Dow loxane (Petrarch Systems); DC-531 siloxane polyamine amines such as 1,3-bis(y-aminopropy))tetramethyl disiides and methacrylamides of siloxane mono, di, or poly- 10 mercaptosuccinic acid, 2,3-dimercaptopropaneaulonic ning); Q4-3557 siloxane diol (Dow Coming); acrylam-(Wacker-Germany); Q4-3667 siloxane diol (Dow Cortrarch Systems, Bristol, Pa.); VP-1610 siloxane diol 1,3-bis(4-hydroxybutyl)tetramethyl disiloxane methacrylates of siloxane mono-, di-, or polyols such as 3 JEFFAMINE TM); mono, di, and polyacrylates and amino or diamino-terminated polyethers (available as thylencoxide glycols (available as TERATHANE TM), groups (available as PLURONICTM), tetrame-

butylaminocthyl methacrylate; allyl methacrylate; dialdimethylaminoethyl acrylate and methacrylate; M-tertand methacrylate; allyl glycidyl ether, acrolein; N,N-(AMPS) and its salts; vinyl azlactones; glycidylacrylate 2-acrylamido-2-methylpropanesulfonic trile; monoallylamine; vinylpyridines; n-vinylpyrrolip-sulfonic acids and their metal salts; 3-aminocrotoniacid and methacrylic acid; vinylsulfonic and styrene tylamide; glyoxal bis-acrylamide; metal salts of acrylic maleimide, M-cyanoethyl acrylamide; M-isopropyl aca-allylether, acrylamide; methacrylamide; maleamide; Jyloxyethanol; o-allyl phenol; divinyl carbinol; glycerol prene; allyl alcohol; allyl glycolate; isobutenediol; allates; maleic anhydride; butadiene; isoprene; chlorolate; hydroxy (C2 to C4) alkyl acrylates and methacry- 35 ide; 2-hydroxy-3-chloropropyl acrylate and methacrymethylolated discetone acrylamide and methacrylamacrylate; diacetone acrylamide and methacrylamide; methylol methactylamide; aziridinyl actylate and methof Formula VI include N-methylol acrylamide; N-R2, or R3. Examples of such functionalized compounds tional group) bonded to the compound through Q, R₁, ble of further elaboration as desired into such a funcplied to a fibrous substrate (or a functional group capaimpart a durable property when the compound is ap- 25 as the adduct of 2-mercaptoethylamine and caprolacmulas I-III that comprise a functional group that can also be used to ultimately provide compounds of Forto linking group Z through Q, R_J, R_Z, or R_J. They can chemical oligomeric portion of a compound is bonded ple, compounds of Formulas I-III wherein the fluoro- 20 also be used in step (1) to ultimately provide, for examsess particular functional groups in G, R5, R6, or R7 can Compounds of Formula V and Formula VI that pos-

zimidazole, 2-mercaptobenzoic acid, 2-mercaptobenzocaptoethanol, mercaptoacetic acid, 2-mercaptobengroups. Examples of such compounds include 2-meror are capable of further transformation into such 65 applied to a fibrous substrate, react with an isocyanate, and others that can render a composition durable when monium groups and salts thereof also discussed above, thereof such as those discussed above, quaternary amoxy, haloformyl, aziridinyl, acid groups and salts 60 end-capping agent include hydroxy, amino, halo, epabove. Suitable functional groups for inclusion in the functionalized end-capping agent L(SH), as defined by way of Scheme I through the use of an appropriate tion, it can be provided (additional) functional groups 55 compounds of Formula V or Formula VI in its preparafunctional groups by virtue of the use of functionalized Whether or not a compound of the invention contains and the like.

DB Name	Query	<u>Hit</u> Count	<u>Set</u> Name
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	110 and benzoic acid [clm]	1	<u>L23</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	110 and benzoic acid [ab]	0	<u>L</u> 22
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	110 and benzoic acid [ti]	0	<u>L21</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	110 and amino benzoic acid	0	<u>L20</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	110 and benzoic acid	54	<u> </u>
USPT, PGPB, JPAB, EPAB, DWPI, TDBD	II0 and acid	438	<u>L18</u>
USPT, PGPB, JPAB, EPAB, DWPI, TDBD	110 and 113 [ti]	11	<u>L</u> 17
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	111 and 113 [ti]	1	<u>L16</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD,	, 111 and 113	21	<u>L15</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	110 and 113	25	<u>L14</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	polyacetal resin or polyoxymethylene resin	4923	<u>L13</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	110 and 111	0	<u>L</u> 12
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	anthranilic acid or 2 adj amino benzoic acid or 4 adj amino benzoic acid	4477	<u>L11</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	formaldehyde near scavenger	617	<u>L10</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	4 adj amino benzoic acid and 3406223 [pn]	0	<u>L9</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	4 adj amino benzoic acid and 5866671 [pn]	0	<u>L8</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	4 adj amino benzoic acid and 5603927 [pn]	0	<u>L7</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	2 adj amino benzoic acid and 5603927 [pn]	0	L6
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	2 adj amino benzoic acid and 5866671 [pn]	0	<u>L.5</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	2 adj amino benzoic acid and 3406223 [pn]	0	Ļ4
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	anthranilic acid and 3406223 [pn]	()	L3
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	anthranilic acid and 5866671 [pn]	0	<u>L2</u>
USPT,PGPB,JPAB,EPAB,DWPI,TDBD	anthranilic acid and 5603927 [pn]	0	<u>L1</u>

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L15: Entry 1 of 2

File: DWPI

Oct 12, 2000

DERWENT-ACC-NO: 2001-024449

DERWENT-WEEK: 200103

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TITLE: New polyacetal resin composition useful in molded articles comprises at least one odor reducing additive e.g. tris(hydroxymethyl)aminomethane or ethyl

p-aminobenzoate

INVENTOR: KASSAL, R J; MORI, H; SHINOHARA, K

PRIORITY-DATA: 1999US-0287432 (April 7, 1999)

PATENT-FAMILY:

PUB-NO

PUB-DATE

LANGUAGE

PAGES MAIN-IPC

WO 200059993 A1

October 12, 2000

018

C08K005/00

INT-CL (IPC): C08K 5/00; C08L 59/00; C08K 5/3445; C08K 5/17; C08K 5/00

Review Classification Date Reference Claims 10000 Draw Desc Image

Document ID: JP 68\(\beta 20\(\beta 08 \) B

L15: Entry 2 of 2

File: DWPI

DERWENT-ACC-NO: 1968-12294Q

DERWENT-WEEK: 196800

COPYRIGHT 2001 DERWENT INFORMATION LTD

TITLE: Polyoxymethylene stabilisation with aliphatic or

PRIORITY-DATA: 1964JP-0025050 (May 4, 1964)

PATENT-FAMILY:

PUB-NO

PUB-DATE

LANGUAGE

PAGES

MAIN-IPC

JP 68020308 B

000

4045+30 108 Classification Date Reference Claims FOMD Orano Desc. Image

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-continued

and the like.

tains a group that can impart soft hand, stain release, water repellency, or a durable property when the compound is applied to a fibrous substrate. A group that can impart stain release is comprised by the fluorochemical impart stain release is comprised by the fluorochemical impart stain release is comprised by the fluorochemical oblgomer portion. In particular with reference to Formulas I-III, any one or more of Q, R₁, R₂, R₃, E, or X can comprise such a group. It is preferred that groups that can impart soft hand, water-repellency, or a durating that can impart soft hand, water-repellency, or a durating that the stain impart soft hand, water-repellency, or a durating that the stain is the stain in the stain in the can impart soft hand.

A fluorinated compound of the invention also con-

ongoiner portion. In particular with reference to Formulas I-III, any one or more of Q, R₁, R₂, R₃, E, or X can comprise such a group. It is preferred that groups that can impart soft hand, water-repellency, or a durable property be comprised by the organic moiety R. Such a group, however, can alternatively be comprised by the fluorochemical oligomeric portion. In particular, with reference to Formulas I-III, any one or more of with reference to Formulas I-III, any one or more of

the substituents Q, R₁, R₂, R₃, R, and E or X can comprise such a group.

Any property that can be imparted through the use of fluorochemicals on fibrous substrates can be imparted

salts thereof with, e.g., the above-listed types of acids. like, or amino and quaternary ammonium groups and thereof, quaternary ammonium salts thereof and the 65 metal and alkaline-earth metal salts thereof, amine salts droxyphosphinyl, and hydroxyphosphinilidene, alkali groups such as carboxy, sulfo, sulfino, sulfeno, dihyamino, hydroxy, halo, haloformyl, aziridino, acid that can undergo a hydrosilation reaction, epoxy, 60 parting durability include polymerizable olefin, olefin the invention. Representative groups suitable for imintended to be treated with the ultimate composition of as a function of the particular fibrous substrate that is groups can be easily selected by those skilled in the art tabric by any of the above-described mechanisms. Such substrate" designates a group that can interact with a property when the compound is applied to a fibrous cation and claims, "a group that can impart a durable polymer chains. For the purposes of the instant specifinon-wovens are essentially non-functional but comprise and carboxamido groups. Other fabrics such as some nylon variously comprise hydroxyl, amino, carboxyl, cause durability. Wool, leather, paper, cotton, and compound and the substrate, or ionic bonding, can free radical or like reactions between the fluorinated bonding by way of nucleophilic, electrophilic, ionic, entanglement, or chemical interaction such as covalent substrate. For example, interactions such as physical interaction between the fluorinated compound and a can be achieved by any of a number of mechanisms of the use of the compositions of the invention. Durability property can be rendered a durable property through by the compositions of the invention, and any such fluorochemicals on fibrous substrates can be imparted

nucleophile with an isocyanate. under conditions conventionally employed to react a groups in R be substantially unreactive to isocyanates polyvalent polysiloxane chain). It is preferred that the 50 polyvalent poly(meth)acrylate chain, or a mono, di, or and combinations thereof (e.g., R can be a mono, di, or taining, sulfur-containing, nitrogen-containing groups, can optionally include oxygen-containing, alicon-conlene, or a mono, di, or polyvalent polymeric chain) and lene, or any combination thereof (e.g., stalky), stalkynyl or alkenylene, aryl or arylene, polyaryl or polyarychain, branched chain, or cyclic alkyl or alkylene, alke-Schemes described in detail below. R can be straight anitable for use in steps (2) and (3) of the Reaction better understood with reference to the compounds structures contemplated for the organic moiety will be (2) of either of the Reaction Schemes. The range of instance where 2,4-toluene dissocyanate is used in step Schemes I and 2 below, or R can be 2,4-tolylene in an isocyanate is used in step (2) of either of Reaction example, R can be methyl in an instance where methyl eynthetic method used to prepare the compound. For group that is present merely as a consequence of the 30 group. In its simplest form, R is a relatively simple Formulas I-III is a mono-, di-, or polyvalent organic The organic moiety, designated R in compounds of and sulfur analogs thereof. Urethanylene is preferred.

The organic moiety K can also contain isocyanate-derived moieties such as those described above in connection with linking group \mathbb{Z} . Further, K can comprise isocyanate-derived moieties that are formed by the reaction of two or more isocyanates with one another, as earbodismidylene ($-N = \mathbb{Z} = \mathbb{Z} = \mathbb{Z}$),

imino-4, 6-dioxohexabydro-1,3,5-oxdiazinylene

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L4: Entry 1 of 11

File: PGPB

Dec 13, 2061

PGPUB-DOCUMENT-NUMBER: 20010051679

PGPUB-FILING-TYPE: new

DOCUMENT-IDENTIFIER: US 20010051679 A1

TITLE: Amino resin mixture for producing foils and edgings with reduced

formaldehyde elimination

PUBLICATION-DATE: December 13, 2001

INVENTOR-INFORMATION:

NAME CITY STATE COUNTRY RULE-47

Scholl, Frank Bad Homburg DE
Wonner, Johann Rodgau DE
Hobisch, Gerald Graz AT

APPL-NO: 09/ 847478 DATE FILED: May 2, 2001

FOREIGN-APPL-PRIORITY-DATA:

COUNTRY APPL-NO DOC-ID APPL-DATE
DE 100 21 849.0 2000DE-100 21 849.0 May 5, 2000

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INT-CL: [07] C08K 3/20, C08F 283/04, C08K 5/36 US-CL-PUBLISHED: 524/211; 524/501, 525/453 US-CL-CURRENT: 524/211; 524/501, 525/453

ABSTRACT:

Amino resin mixture for producing foils and edgings with reduced formaldehyde elimination Aqueous amino resin compositions comprising amino resins A, formaldehyde-binding additives (auxiliaries) B, which may include hydroxyl-containing polyurethanes B13, acrylic resins C, if desired, in the form of aqueous dispersions, and water, and their use as impregnating compositions for paper for the purpose of producing finished foils and edgings.

FIELD OF THE INVENTION

[0001] The present invention relates to aqueous, heat-curable amino resin mixtures based on etherified amino resins and formaldehyde-binding auxiliaries and to their use for impregnating papers and cardboard.

BACKGROUND OF THE INVENTION

[0002] Surfaces and narrow faces of woodbase materials are coated using coated foils (finished foils) and, respectively, impregnated cardboard (Kunststoff-Handbuch Vol. 10 Duroplaste [Thermosets], Hanser-Verlag, 2nd Ed. 1988, p. 462 f., pp. 477 to 479). For the production of finished foils (to coat surfaces) and finished edgings (to coat narrow faces, of boards in particular), absorbent papers are impregnated with solutions or dispersions of (thermosetting) amino resins such as urea-formaldehyde and/or melamine formaldehyde resins, for example, alone or in combination with dispersions of thermoplastics such as acrylic dispersions or styrene-acrylic dispersions, dried in a stream of hot air and simultaneously cured, and then coated.

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Page 2

Application/Control Number: 09/066275

HTT : tinU nA

1. A request for extension of time (paper no. 8) and an amendment (paper no. 9) have been

filed on May 11, 2000.

2. Applicant's election with traverse of claims 1-9 are in Paper No. 9 is acknowledged. The traversal is on the ground(s) that it would not constitute an undue burden to search all the subject matter as claimed. This is not found persuasive because the applicants have made only a

conclusionary statement without any support for it. There <u>is</u> actually an undue burden for search for non-elected claim 10.

The requirement is still deemed proper and is therefore made FINAL.

3. Rejection of claim under 35 U.S.C. 112, second paragraph (see section 3, page 3 of office

action paper no. 5) is now withdrawn.

4. The text of those sections of Title 35, U.S. Code not included in this action can be found

Claims 1-9 and (newly added) 11-14 are rejected under 35 U.S.C. 103(a) as being

unpatentable over Schlumpf (Canadian Patent 2016447) in view of Shaw et al (USP 4643940),

Hosoda et al (USP 3812225) and Figjii et al (USP 4680318).

This rejection (also to be applied to new claims 11-14) is incorporated here by reference

from section 4, pages 3-5, of prior office, paper no. 5).

Applicant's arguments filed May 11, 2000 (paper no. 9) have been fully considered but

they are not persuasive.

in a prior Office action.

[0003] In order to ensure adequate penetration of the impregnation liquors into the paper or cardboard, the resin solutions must be processed from aqueous or alcohol dilution. Owing to the high level of cellulose swelling in aqueous systems, the foils and edgings produced from high aqueous dilution are brittle, exhibit a high level of water absorption, and even in the coated state have a surface whose visual appeal is low. The procedure described in DE-A 23 09 334, comprising impregnating liquors diluted with C.sub.1 to C.sub.4 alcohols, does give foils and edgings having good performance properties but requires complex measures for reprocessing the waste gas. The route to a solution that is described in DE-A 44 39 156, modifying melamine resins with guanamines, makes it possible to carry out impregnation from purely aqueous impregnating liquors. A disadvantage, however, is the significantly higher cost of the amino resin, resulting from the use of the guanamines.

[0004] The formaldehyde emissions of finished foils and edgings after manufacturing are determined following storage under standard climatic conditions (23.degree. C., 50% relative atmospheric humidity) in accordance with the standard EN 717-2 (FESYP method, gas analysis). Foils and edgings with values of less than 3.6 mg/(h.multidot.m.sup.2) by the FESYP method meet the limit of the standard ("E 1"). The rates of emission found remain more or less constant even after several weeks of storage under standard climatic conditions. Formaldehyde emissions observed on the films and edgings arise due to the use of urea-formaldehyde and/or melamine-formaldehyde resins in the impregnating liquors for impregnating the paper or cardboard sheets and/or in the coating formulations for coating the films and edgings. By using particularly low-formaldehyde urea-formaldehyde and/or melamine-formaldehyde resins (with low formaldehyde clearage) it is possible to reduce the formaldehyde emissions as measured by the FESYP method (standard climatic conditions) to values around 2 mg/(h.multidot.m.sup.2).

[0005] It has surprisingly now been found that when edgings produced in accordance with the prior art and originally (before the commencement of storage) satisfying the classification E1 ("E1 edgings"), with initial formaldehyde emission values of from 1.0 to 3.5 mg/(h.multidot.m.sup.2), are stored under nonstandardized climatic conditions, at customary summer temperatures and atmospheric humidities, the formaldehyde emissions rise in the course of a few weeks to values of in some cases much higher than 3.5 mg/(h.multidot.m.sup.2), and so the edgings no longer meet the E1 criterion. This unexpectedly high increase in the formaldehyde emissions was confirmed by storage under defined conditions in a tropical climate (35.degree. C., 90% relative atmospheric humidity), with measurement being carried out only after 3-day reconditioning under standard climatic conditions following storage under the tropical climatic conditions.

[0006] From the prior art it is known that the amount of free formaldehyde and also the formaldehyde emissions may be reduced by adding formaldehyde scavengers such as urea and urea derivatives, for example. For instance, according to DE-A 38 37 965, finished foils and edgings with formaldehyde emissions that are negligible as determined in accordance with DIN 52368 may be produced by adding urea to the melamine-formaldehyde condensation product. Regarding the behavior during and after storage under tropical climatic conditions, however, no information is given. According to DE-A 34 03 136, mixtures of organic hydroxy compounds and an amide are suitable for use as formaldehyde-binding agents in boards made from wood cellulose materials. The use of these mixtures as formaldehyde scavengers in finished foils and edgings is not described. The addition of the mixtures described in DE-A 34 03 136 to amino resins that are used to produce finished foils and edgings leads to a marked deterioration in the flexibility of the finished foils and edgings produced with them. The use of formaldehyde scavengers known from the literature, such as urea, ethyleneurea and propyleneurea, resulted in finished foils and edgings which met the El criterion under standard climatic conditions but which markedly exceed the E1 limit of 3.5 mg/(h.multidot.m.sup.2) under tropical climatic conditions.

[0007] It is therefore an object of the present invention to develop amino resinmixtures for producing films and edgings, which can be used to produce, relative to the prior art, finished foils and furniture edgings with significantly reduced formaldehyde emission when stored under tropical climatic conditions, while retaining the required performance properties.

SUMMARY OF THE INVENTION

Applicant's arguments do not comply with 37 CFR LIII(c) because they do not clearly point out the patentable novelty which he or she thinks the claims present in view of the state of the art disclosed by the references cited or the objections made. Further, they do not show how the amendments avoid such references or objections.

On page 6, paragraph 2 of above response paper no. 9, the applicants argue that Schlumpf

teaches away from claimed invention because Schlumpf discloses solids at lease 92.1%. While there is a difference of only 0.1% (between claimed 92.0% and disclosed 92.1%), this difference fails to distinguish patentably the instant invention from the prior art, particularly because the applicants have not established the criticality of the upper limit viz. 92.0%.

Applicants' further argue in the next paragraph on the same page, that Schlumpf does not

teach claimed particle size distribution this argument is not persuasive. On page 9, Schlumpf discloses a calcium carbonate of a mean statistical particle diameter of 0.5 - 50 microns. This range overlaps that of calcium carbonate (viz. 0.3 to 18 microns) in example 1 in instant specification page 14. Furthermore, also the applicants have not shown any criticality of having the only the claimed particle size distribution.

cannot degas the composition and therefore Schlumpf teaches away form in inclusion of a blowing agent. Wether the apparatus can or cannot degas the composition has no influence at all.

On page 6, paragraph 4 of same paper, the applicants state that apparatus of Schlumpf

[0014] Likewise suitable as formaldehyde-binding component B2 are reaction products containing urethane groups, said products being obtained by reacting polyhydroxyl compounds B21, including the compounds mentioned under B12 and also aliphatic polyhydroxy amines B211 having from 2 to 6 hydroxyl groups and 1 to 4 nitrogen atoms, attached in an aminelike manner, per molecule and containing no free amine-type hydrogen atoms, such as N-methyldiethanolamine, N,N,N',N'-tetrakis(2-hydroxyethyl)ethylen- ediamine and triethanolamine with monofunctional or polyfunctional aliphatic, cycloaliphatic or aromatic isocyanates B22, such as hexamethylene diisocyanate, for example.

[0015] The addition of formaldehyde-binding auxiliary B (calculated by mass without solvents or diluents, i.e., on a 100% basis) amounts to from 2 to 50 parts by weight per 100 parts by weight of amino resin (likewise on a 100% basis). In addition to the reduction in formaldehyde, it is also found when using a reaction product of hydroxy amines and diisocyanate, such as the reaction product of triethanolamine with hexamethylene diisocyanate in Example 11, that the pot life is extended significantly.

[0016] The acrylic resin dispersion C is a dispersion of an acrylic copolymer in water, preparable for example by emulsion copolymerization of olefinically unsaturated monomers, the monomer mixture used for its preparation comprising a predominant fraction (more than 50% of its mass) of what are known as acrylic monomers, i.e., acrylic or methacrylic acid and derivatives thereof, especially esters with aliphatic alcohols having 1 to 10 carbon atoms, esters with aliphatic polyhydroxy compounds having 2 to 10 carbon atoms and at least two hydroxyl groups per molecule, and the nitriles of said acids. Preferred acrylic monomers among the esters are methyl, ethyl, n-butyl, t-butyl, hexyl and 2-ethylhexyl (meth)acrylate, hydroxyethyl and hydroxypropyl (meth) acrylate. It is additionally possible for copolymerizable monomers such as styrene and other aromatic vinyl compounds, esters or monoesters of olefinically unsaturated dicarboxylic acids such as, in particular, maleic acid, vinyl esters such as vinyl acetate or vinyl Versatate, vinyl halides or vinyl ethers to be copolymerized. The synthetic resin dispersions usually have mass fractions of solids of from 25 to 85%; they are added to the amino resin or else to the impregnating liquor itself. The ratio of the mass of the acrylic copolymer in the acrylic dispersion to the mass of the amino resin in its aqueous solution or dispersion may within the composition be from 0 to 150:100, preferably from 20 to 140:100.

[0017] The amino resin compositions are used to impregnate absorbent papers or cardboards. The amount of amino resin composition introduced is usually such that the mass per unit area of the paper or cardboard following impregnation and subsequent drying increases by a factor of from 1.3 to 2.5, preferably from 1.4 to 1.8.

[0018] The use of the mixtures of the invention leads to a significant reduction in the formaldehyde emissions from the foils and edgings stored under tropical climatic conditions.

EXAMPLES

[0019] Preparation of a Partially Etherified Melamine-formaldehyde (MF) Resin

[0020] A 30 l laboratory vessel with stirrer, reflux condenser and thermometer was charged with 6717 q (87.2 mol) of 39% strength aqueous formaldehyde and this initial charge was heated to 68.degree. C. Then 31.5 ml of 2 N sodium hydroxide solution were added followed immediately by 3450 g (27.4 mol) of melamine. Because of the exothermic reaction of melamine and formaldehyde, the mixture rose in temperature to about 83.degree. C. and was held at this temperature until all of the melamine had dissolved. It was then cooled to 55.degree. C. and 16560 g (517 mol) of methanol and 30 ml of 53% strength nitric acid were added. The reaction mixture was heated to 59.degree. C. and stirred at this temperature until a clear solution was formed. After a further 30 minutes, the reaction was terminated by adding about 140 ml of 2 N NaOH. The pH was adjusted to 10. Excess methanol was removed by distillation under reduced pressure (generated by a water jet pump) and the mass fraction of solids of the resin was adjusted to 75% (measured on a 2 g sample, dried at 120.degree. C. for 1 h in a glass dish). The resin had the following characteristics: content (mass fraction of solids): 75%; viscosity at 23.degree. C.: 480 mPa.multidot.s, mass fraction of free formaldehyde: 0.17%;

2216-5441 []

Application/Control Number: 09/047161

HYI : JinU nA

1. The request filed on May 11, 2000 (paper no. 15) for a Continued Prosecution Application (CPA) under 37 CFR 1.53(d) based on parent Application No. 09/047161 is acceptable and a CPA has been established. An action on the CPA follows.

Claims 1-16 are pending, out of which claim 15 & 16 are non-elected, leaving 1-14 to be

examined.

2. Rejection of claim 1 under 35 U.S.C. 102(b) set forth in section 3, pages 3 & 4 of office action of January 25, 1999 (paper no. 5) and maintained in later office actions, is now withdrawn as a result of amendment. That of claim 13 is however maintained.

3. The text of those sections of Title 35, U.S. Code not included in this action can be found in a prior Office action.

Claims 1-14 are rejected under 35 U.S.C. 103(a) as being unpatentable over Taubitz et al

(USP 4774286) in view of Levine et al (USP 4321087), Kohara et al (USP 5302656) and Clark et al (USP 5510398).

(All references are of record in prior office actions).

and a graft copolymer (abstract). The graft copolymer is based on acrylic acid esters, methacrylic acid esters and/or conjugated dienes and an epoxy-containing monomer (col. 3, lines 1-12). Elastomeric consistuents viz rubbers can be included also (col. 4, lines 3-13). (These rubbers read on those of instant claim 4). Metal flakes and metal fibers and mineral particles are also added to such molding material (col. 4, lines 44-50 and 60-63).

Tanbitz discloses thermoplastic molding materials based on polycarbonates, polyesters

[0008] The invention provides aqueous amino resin compositions comprising amino resins A, formaldehyde-binding additives (auxiliaries) B, which may comprise hydroxyl group-containing polyurethanes B13, and, if desired, acrylic resins (in the form of aqueous dispersions, and water.

DETAILED DESCRIPTION OF THE INVENTION

[0009] Where the component B consists only of at least one organic hydroxyl compound which is soluble in water or a monohydric alcohol having 1 to 4 carbon atoms and is selected from dihydric, trihydric and pentahydric alcohols containing up to 6 carbon atoms, pentaerythritol and sorbitol, monosaccharides containing up to 6 carbon atoms, disaccharides containing up to 12 carbon atoms, polysaccharides having an Ostwald viscosity of up to 200 mPa.multidot.s at 25.degree. C. and a concentration corresponding to 37% refraction, monohydric and polyhydric aromatic alcohols containing only one benzene ring, and monohydric and polyhydric phenols, and of at least one amide which is soluble in water or a monohydric alcohol having 1 to 4 carbon atoms and is selected from aliphatic amides containing up to 6 carbon atoms and aromatic amides containing only one benzene ring, the presence of at least one of the components B13 and C in the composition is mandatory.

[0010] The amino resins A are water-soluble melamine resins, urea resins or mixed melamine-urea condensates which are etherified with C.sub.1 to C.sub.4 alcohols. In the compositions of the invention it is also possible to use those melamine resins in which a fraction (up to 20% of its mass) of the melamine has been replaced by other triazines such as acetoguanamine, caprino-guanamine or benzoguanamine. Preferred resins, however, contain less than 10%, in particular less than 5%, of other triazines, measured on the same scale. Particular preference is given to straight melamine resins or to their cocondensates with urea. The resins are etherified at least partially with the abovementioned alcohols, especially methanol, n-butanol and isobutanol. Particular preference is given to methanol-etherified amino resins.

[0011] Particular preference is given above all to melamine resins having an amount-of-substance ratio (molar ratio) of melamine to urea to formaldehyde to methanol of 1 mol: $(0\ to\ 2\ mol):(0.8\ to\ 5.8\ mol):(0.8\ to\ 5.5\ mol)$.

[0012] The preparation of the amino resins A is widely known. First of all, methylolation and condensation are carried out by adding formaldehyde to the amino resin formers at pH values from 7 to 10 and temperatures from 40 to 110.degree. C., after which the etherifying alcohol is added and reaction is continued at pH values from 1 to 7 and temperatures from 30 to 80.degree. C. The condensation conditions and etherification conditions are guided by the water dilutability desired for the resin, which amounts to at least 1 part by weight of resin to 5 parts by weight of deionized water, and by the required penetration properties.

[0013] As component B, formaldehyde-binding auxiliaries are added. These auxiliaries are selected from mixtures B1 of organic amides B11 having up to 19 carbon atoms and from one to four nitrogen atoms attached in amidelike or imidelike manner, such as urea and/or urea derivatives such as thiourea, ethyleneurea (2-imidazolidinone), propyleneurea, acetyleneurea (glycoluril), and also formamide, acetamide, benzamide, oxalamide, succinimide, malonamide and dicyandiamide, and polyhydroxyl compounds B12 selected from aliphatic linear and branched compounds B121 having from 2 to 6 hydroxyl groups and 1 to 10 carbon atoms, such as glycol, 1,2- and 1,3-propylene glycol, neopentyl glycol, glycerol, trimethylolpropane, ditrimethylolpropane, erythritol, pentaerythritol, dipentaerythritol, sorbitol and mannitol, monosaccharides B122 having up to 6 carbon atoms, and disaccharides B123 having up to 12 carbon atoms, and, if desired, water-soluble or water-dispersible, hydroxyl-containing urethane compounds B13. These urethane compounds are of low mol mass (number-average molar mass M.sub.n from 150 to 5000 g/mol, preferably from 300 to 4000 g/mol) and contain hydrophilic groups which are preferably nonionic, especially building blocks derived from glycol or from oligoethylene or polyethylene glycol. Examples of suitable compounds are adducts of aliphatic linear or cyclic diisocyanates, such as 1,2-diisocyanatoethane and 1,6-diisocyanatohexane, with ethylene glycol, diethylene glycol or mixtures thereof with 1,2- or 1,3-propylene glycol, these latter hydrophobic diols being used only in fractions (e.g., less than 25% of the mass of diols overall) such that the adduct remains soluble or dispersible, respectively, in water.

1171 :tinU mA

Any inquiry of a general nature or relating to the status of this application or proceeding

should be directed to the receptionist whose telephone number is (703) 308-0661.

U.K. Rajguru/om August 14, 2000 water dilutability: unlimited.

[0021] Auxiliary BA: Mixture of Glycerol and Urea

[0022] 100 g of urea were introduced with stirring into 100 g of glycerol and the mixture was heated to 90.degree. C. After the urea had dissolved, it was cooled to 20.degree. C. This gave a mixture with a pastelike consistency.

[0023] Auxiliary BB: Mixture of Glycerol, Urea and Polyurethanediol

[0024] 100 g of urea and 67 g of 88% strength solution of urethanediol (number-average molar mass M.sub.n 320 g/mol, OH number 350 mg/g, urethane group content 37 cg/g) were introduced with stirring into 100 g of glycerol and the mixture was heated to 90.degree. C. Following dissolution, the mixture was cooled to 20.degree. C. This gave a mixture with a pastelike consistency.

[0025] Auxiliary BC: Reaction Product of Trimethylolpropane (TMP) and Hexamethylene Diisocyanate (HDI)

[0026] In a suitable reaction vessel with water separator, 100 g of TMP were melted, after which nitrogen was passed over the material at 140.degree. C. with stirring for 3 hours in order to eliminate traces of water. The system was then cooled to 60.degree. C. and 8 g of HDI was added slowly dropwise with vigorous stirring. The temperature was maintained until the mass fraction of isocyanate groups in the reaction product (NCO value) had fallen below 0.1%. Then the mass fraction of solids was adjusted to 85% using water. The reaction mixture was a colorless solution of low viscosity.

[0027] Auxiliary BD: Reaction Product of Tripropylene Glycol and Hexamethylene Diisocyanate

[0028] In a suitable reaction vessel with water separator, 100 g of tripropylene glycol were introduced, after which nitrogen was passed over the material at 140.degree. C. with stirring for 3 hours in order to eliminate traces of water. The system was then cooled to 40.degree. C. and 5 g of HDI were added slowly dropwise with vigorous stirring. The temperature was maintained until the NCO value had fallen below 0.1%. Then the mass fraction of solids was adjusted to 85% using water. The reaction mixture was a pale yellow solution of low viscosity.

[0029] Auxiliary BE: Reaction Product of Glycerol and Hexamethylene Diisocyanate

[0030] In a suitable reaction vessel with water separator, 100 g of glycerol were introduced, after which the water was removed azeotropically at 140.degree. C. for 5 hours using special boiling-point spirit 80/120 as azeotrope former. Following the removal of the azeotrope former (by distillation), the system was then cooled to 60.degree. C. and 5 g of HDI were added slowly dropwise with vigorous stirring. The temperature was maintained until the NCO value had fallen below 0.1%. The reaction mixture was a yellow solution of medium viscosity.

[0031] Auxiliary BF: Reaction Product of Triethanolamine and Hexamethylene Diisocyanate

[0032] In a suitable reaction vessel with water separator, 100 g of triethanolamine were introduced, after which the water was removed azeotropically at 140.degree. C. for 5 hours using special boiling-point spirit 80/120 as azeotrope former. Following the removal of the azeotrope former, the system was then cooled to 40.degree. C. and 15 g of HDI were added slowly dropwise with vigorous stirring. The temperature was maintained until the NCO value had fallen below 0.1%. The reaction mixture was a yellow solution of medium viscosity.

[0033] Performance Testing:

[0034] The MF resin described above was used in each of the examples. The acrylic dispersion used was a dispersion based on a copolymer of methyl methacrylate, butyl acrylate, hydroxyethyl methacrylate, acrylic acid and styrene, having a hydroxyl number of about 120 mg/g and a mass fraction of solids of about 50%, which was diluted if necessary to the lower specified value (45%).

Inventive Examples 1-4 and Comparative Examples 1-4

Tanbitz does not mentioned the (claimed) particle size of metallic particles and mineral

particles, average diameter and average aspect ratio of fibers.

Levine discloses that a preferred metallic pigment should have a diameter of 10-20

Kohara discloses that the average diameter of a fibrous filler is 0.1 to 100 micrometers,

microns (see Levine, col. 5, lines 30-34).

filler is 0.01 micrometer to 1 millimeter (see Kohara, col. 4, lines 27-38).

and the length is from 5 micrometers to 20 millimeters. Average article diameter of powdery

Clark discloses thermoplastic compositions having a speckled surface appearance; This

appearance results form the use of nondispersing pigments (abstract; col. 10, lines 1-2). It is

preferred that these pigments possess a large aspect ratio greater than about 20 (col. 10, lines 33-

.(85

`t

Based on the teachings of Levine, Kohara and Clark, it is the examiner's position that it

would have been obvious to one ordinary skill in the art to use in the composition of Taubitz

metallic particles of claimed particle size, fibers of claimed average diameter and average aspect

ratio and interala particles of claimed average particle size in order to produce products having

bright reflective appearance, enhanced hiding power; improved crack resistance and moldability

as well as better speckled appearance.

Any inquiry concerning this communication or earlier communications from the

examiner should be directed to U.K. Rajguru whose telephone number is (703) 308-3224. The

examiner can normally be reached on Monday - Friday from 9:30 am to 6:00 pm.

[0035] 130 g of 50% acrylic dispersion and 1.7 g of p-toluenesulfonic acid were added in each case to 100 g of the above-described MF resin, along with the auxiliaries indicated in Table 1 for the individual application examples. Following dilution to a liquor concentration of 50%, each of these liquors was used to impregnate papers having a mass per unit area of 200 g/m.sup.2 (typical edgebanding cardboard) and the impregnated edgings were dried at 160.degree. C. to a residual moisture content of about 2%. Following impregnation and drying, the basis weight was about 330 g/m.sup.2. The impregnated edgings obtained in this way were coated with an aqueous acid-curing varnish (plasticized urea resin; combination of a urea resin with a short- to medium-oil alkyd resin, with p-toluenesulfonic acid as curing agent) and dried at a temperature of 160.degree. C. to a residual moisture content of 1.5%. The varnish addon was about 20 g/m.sup.2. With regard to their performance properties, each of the edgings obtained met the requirements.

[0036] To determine the formaldehyde emissions, the edging samples were conditioned for 3 days under standard climatic conditions (23.degree. C., 50% relative atmospheric humidity) prior to each measurement. To determine the formaldehyde emissions under tropical climatic conditions, the edgings were stored for 1 to 4 weeks at 35.degree. C. and 90% relative atmospheric humidity, after which they were reconditioned for 3 days under standard climatic conditions, prior to the actual measurement. The formaldehyde emissions were measured in accordance with EN 717-2. Table 1 lists the resulting formaldehyde emissions in mg/(h.multidot.m.sup.2) as averages of the 1- to 4-hour values:

1 TABLE 1 Directly after After After After After Auxiliary prep. 1 wk 2 wks 4 wks 6 wks Inventive 4 g BA 1.7 4.5 3.8 3.6 3.3 Example 1 Inventive 12 g BA 0.8 3.6 3.2 2.8 2.1 Example 2 Inventive 4 g BB 1.4 3.9 3.4 2.5 2.3 Example 3 Inventive 12 g BB 1.1 3.5 3.0 2.3 2.2 Example 4 Comp. Ex. 1 -- 2.0 5.2 4.4 3.7 4.2 Comp. Ex. 2 5 g urea 1.9 5.1 4.2 4.1 4.0 Comp. Ex. 3 12 g urea 1.4 5.4 3.8 3.2 2.6 Comp. Ex. 4 12 g glycerol 1.6 4.6 3.7 3.4 2.5

[0037] The edgings produced in accordance with Inventive Examples 2 and 4 meet the E1 criterion (i.e., 3.6 mg/h/m.sup.2) even after one week of storage under tropical climate conditions.

Inventive Example 5 and Comparative Example 5

[0038] 1.5 g of p-toluenesulfonic acid were added to 100 g of the above-described MF resin, along with the auxiliaries indicated in Table 2 for the individual application examples. These undiluted liquors were used to impregnate papers (typical edgebanding cardboard; 200 g/m.sup.2) by means of knife application from the decorative side, and the impregnated edgings were dried at 160.degree. C. to a residual moisture content of about 1.5%. Thereafter, the basis weight was about 305 g/m.sup.2. The impregnated edgings thus obtained were coated with an aqueous acid-curing varnish (see above) and dried at a temperature of 160.degree. C. to a residual moisture content of 1.5%. The varnish addon was about 20 g/m.sup.2. With regard to their performance properties, the edgings obtained in each case met the requirements.

[0039] The formaldehyde emissions (reported in mg/(h.multidot.m.sup.2) were determined as in Example 1.

2 TABLE 2 Directly after After After After After Auxiliary prep. 1 wk 2 wks 4 wks 6 wks Inventive 12 g BB 1.0 2.2 2.1 1.8 1.8 Example 5 Comp. Ex. 5 -- 1.0 3.3 2.4 2.9 2.9

Inventive Examples 6 and 7 and Comparative Examples 6 and 7

[0040] 200 g of 50% acrylic dispersion and 1.7 g of p-toluenesulfonic acid were added to 100 g of the above-described MF resin, along with the amounts of auxiliaries indicated in Table 3 for the individual application examples and also PEG 400 (polyethylene glycol having a number-average molar mass M.sub.n of about 400 g/mol). Following dilution to a liquor concentration of 47% (mass fraction of the resins in the aqueous liquor), these resins were used to impregnate papers (typical edgebanding cardboard; mass per unit area about 200 g/m.sup.2) and the impregnated edgings were dried at 170.degree. C. to a residual moisture content of about 1.6%. Following impregnation, the final weight was about 330 g/m.sup.2. The

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Jim Seidleck, can be reached on (703) 308-2462. The fax phone number for the organization where this application or proceeding is assigned is (703) 305-3599.

Any inquiry of a general nature or relating to the status of this application or proceeding

should be directed to the receptionist whose telephone number is (703) 308-0661.

M.K. Rajguru/om August 14, 2000

conditioned for 3 days under standard climatic conditions (23.degree. C., 50% relative atmospheric humidity) prior to each measurement. To determine the formaldehyde emissions under tropical climatic conditions, the edgings were stored for 1 week at 35.degree. C. and 90% relative atmospheric humidity, after which they were reconditioned for 3 days under standard climatic conditions, prior to the actual measurement. The formaldehyde emissions were measured in accordance with EN 717-2. Table 5 lists the resulting formaldehyde emissions as averages of the 1- to 4-hour values. The flexibility of the edgings was assessed at room temperature with the aid of the flexural test. The parameter reported was the band radius at which the edging still just did not fracture. As is evident from Table 5, the formaldehyde emissions can be reduced significantly relative to Comparative Example 10 while retaining a very low band radius. The desired formaldehyde reduction cannot be achieved by adding small amounts of urea and sorbitol (Comparative Example 11). The pot life is the time taken for the impregnating liquor, stored at 30.degree. C., to obtain a viscosity (measured as the efflux time in accordance with DIN 53211 at 23.degree. C.) of more than 60 seconds or for the penetration time with a defined test paper to rise to more than 70 seconds. The auxiliary BF has a considerable advantage as compared with the prior art, with regard to formaldehyde emissions and pot life.

5TABLE 5 Pot Directly life at Band after After 30.degree. C. radius Auxiliary 100% PEG 400 Sorbitol Urea prep. 1 wk in h in mm Inventive 9g BE 27 g 1.1 g 1.1 g 0.46 1.47 8 6 to Example 10 8 Inventive 9g BF 27 g 1.1 g 1.1 g 0.17 0.46 >30 5 to Example 11 6 Comp. Ex. 10 -- 36 g -- -- 0.75 1.98 6 5 to 6 Comp. Ex. 11 -- 36 g 1.1 g 1.1 g 0.58 1.82 7 5 to 6

CLAIMS:

- 1. An aqueous amino resin composition comprising amino resins A, formaldehyde-binding additives (auxiliaries) B, which if desired comprise hydroxyl-containing polyurethanes B13, if desired, acrylic resins C in the form of aqueous dispersions, and water, with the proviso that the presence of at least one of the components B13 and C in the composition is mandatory where the component B consists only of at least one organic hydroxyl compound which is soluble in water or a monohydric alcohol having 1 to 4 carbon atoms and is selected from dihydric, trihydric and pentahydric alcohols containing up to 6 carbon atoms, pentaerythritol and sorbitol, monosaccharides containing up to 6 carbon atoms, disaccharides containing up to 12 carbon atoms, polysaccharides having an Ostwald viscosity of up to 200 mPa.multidot.s at 25.degree. C. and a concentration corresponding to 37% refraction, monohydric and polyhydric aromatic alcohols containing only one benzene ring, and monohydric and polyhydric phenols, and of at least one amide which is soluble in water where a monohydric alcohol having 1 to 4 carbon atoms and is selected from aliphatic amides containing up to 6 carbon atoms and aromatic amides containing only one benzene ring.
- 2. The aqueous amino resin composition as claimed in claim 1, wherein the amino resins A are water-soluble melamine resins, urea resins or melamine-urea cocondensates which are etherified with C.sub.1 to C.sub.4 alcohols.
- 3. The aqueous amino resin composition as claimed in claim 1, wherein the amino resins A are methanol-etherified melamine resins or melamine/urea resins having an amount-of-substance ratio (molar ratio) of melamine to urea to formaldehyde to methanol of 1 mol:(0 to 2 mol):(1.8 to 5.8 mol):(0.8 to 5.5 mol).
- 4. The aqueous amino resin composition as claimed in claim 1, wherein the ratio of the mass of component B to the mass of the amino resin A in the mixture is from 1 to 30:100.
- 5. The aqueous amino resin composition as claimed in claim 1, wherein the formaldehyde-binding auxiliaries B are selected from mixtures Bi of organic amides Bil having up to 10 carbon atoms and from one to four nitrogen atoms, attached in amidelike or imidelike manner, and polyhydroxyl compounds Bil selected from aliphatic linear and branched compounds Bill having from 2 to 6 hydroxyl groups and 1 to 10 carbon atoms, monosaccharides Bill having up to 6 carbon atoms, and disaccharides Bill having up to 12 carbon atoms, and, if desired, water soluble or water dispersible, hydroxyl-containing urethane compounds Bill.
- 4. The aqueous amino resin composition as claimed in claim 1 or 5, wherein urethane compounds B13 are of low molecular mass, having an above average molar

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mass M.sub.n of from 150 to 5000 g/mol, and contain hydrophilic groups.

- 7. The aqueous amino resin composition as claimed in claim 1, wherein formaldehyde-binding auxiliaries B used comprise the components B2, which are obtainable by reacting polyhydroxyl compounds B21 selected from aliphatic linear and branched compounds B121 having from 2 to 6 hydroxyl groups and 1 to 10 carbon atoms, monosaccharides B122 having up to 6 carbon atoms, and disaccharides B123 having up to 12 carbon atoms, and also aliphatic aliphatic polyhydroxy amines B211 having from 2 to 6 hydroxyl groups and 1 to 4 nitrogen atoms, attached in an aminelike manner, per molecule, and containing no free amine-type hydrogen atoms, with monofunctional or polyfunctional aliphatic, cycloaliphatic or aromatic isocyanates B22.
- 8. The aqueous amino resin composition as claimed in claim 1, wherein the acrylic resins C are aqueous dispersions of an acrylic copolymer in water, the parent monomer mixture containing more than 50% of its mass of acrylic monomers selected from acrylic acid and methacrylic acid, their esters with aliphatic alcohols having 1 to 10 carbon atoms, their esters with aliphatic polyhydroxy compounds having 2 to 10 carbon atoms and at least two hydroxyl groups per molecule, and the nitrites of said acids.
- 9. The aqueous amino resin composition as claimed in claim 8, wherein the ratio of the mass of the acrylic copolymer to the mass of the amino resin in the mixture is from 0 to 150:100.
- 10. The use of the aqueous amino resin composition as claimed in claim 1 for impregnating paper or cardboard for use as finished foils or edgings.
- 11. A finished foil or edging comprising cardboard or paper impregnated with the aqueous amino resin composition as claimed in claim 1.
- 12. The finished foil or edging as claimed in claim 11, wherein the mass per unit area of the paper or cardboard following impregnation and subsequent drying is greater by a factor of from 1.3 to 2.5 than that of the substrate that is used.

25 Perfluorinated aliphatic groups (i.e., those of the for-CP3CF2CF2-, (CF3)7CF-, -CF2SF5, or the like. ably contains at least 7 fluorine atoms, e.g., Phorinated terminal group. This terminal group prefer-The terminal portion of the Rygroup contains a fully preferably about 50% to about 78% fluorine by weight. about 40% to about 78% fluorine by weight, more valent sulfur, or mitrogen. It is preferred that Rycontain catenary heteroatoms such as oxygen, divalent or hexaizable olefinic unsaturation and can optionally contain cyclic alkylene groups. Ryis preferably free of polymertions thereof with straight chain, branched chain, or alkylene groups or combinations thereof or combinastraight chain, branched chain, or cyclic fluorinated ably about 6 to about 14 carbon atoms. Rycan contain preferably 3 to about 20 carbon atoms, and most prefercrably contains at least about 3 carbon atoms, more ety which is both oleophobic and hydrophobic. Reprefinert, non-polar, preferably saturated, monovalent moifluorine being essentially in the Regroups. Reis a stable, on the total weight of the compound, the loci of the 25 percent to about 55 percent fluorine by weight based percent to about 65 percent, and most preferably about cent to about 80 percent, more preferably from about 20

mula CnF2n+1) are the most preferred embodiments of

10:1 to about 2:3, and most preferably about 4:1. mer be from 20:1 to about 1:4, preferably from about polymerized units derived from non-fluorinated monoderived from fluorochemical monomer to moles of that the overall ratio of moles of polymerized units tion of a composition of the invention. It is preferred ily by the relative amounts thereof used in the preparanon-fluorinated monomer, which is determined primarchemical monomer to polymerized units derived from overall ratio of polymerized units derived from fluorothe invention can be characterized with respect to the tion. Therefore, such compositions and compounds of pound present in a particular composition of the invenfluorinated monomer will not be the same in each commoles of polymerized units derived from nonixed units derived from fluorochemical monomer to preferably about 4 units. The ratio of moles of polymerbackbone preferably comprises from 2 to about 8, most Iluorinated monomers are used, likewise the aliphatic 40 instances where both fluorochemical and nonsbout 8, most preferably about 4, polymerized units. In ferred that the aliphatic backbone comprise from 2 to fluorochemical monomers are present, it is more premonomers. In instances where only units derived from 35 20 polymerized units derived from non-fluorinated organic group Ryas defined above), and from 0 to about monomers (i.e., monomers containing a fluorinated about 20 polymerized units derived from fluorinated aliphatic backbone preferably comprises from 2 to 30 merized units to render the portion obgomeric. The mene bornon combuses a sufficient number of poly-The aliphatic backbone of the fluorochemical oligo-

derived linking groups Z include dimer, trimer, oligomer, or the like. Suitable isocyanateamine, or a thiol with an isocyanate or an isocyanate the reaction of a nucleophile such as an alcohol, an isocyanate-derived linking group that can result from in the formulas used herein. Z is a non-polymeric the organic portion by a linking group designated as Z The fluorochemical oligomeric portion is linked to

> property when the compound is applied to a fibrous 15 soft hand, stain release, water repellency, or a durable R1, R2, R3, Q, and X comprises a group that can impart compound of Formula IIIA, optionally at least one of the compound is applied to a fibrous substrate. In a release, water repellency, or a durable property when 10 E comprises a group that can impart soft hand, stain IA or IIA, optionally at least one of R1, R2, R3, Q, and m, a, b, R, R, E, Q, R, R, X, and Z are as defined above for Formulas 1-111. In a compound of Formulas (n+q)-valent organic group such as 2,4-tolylene; and n, wherein q is an integer from 1 to about 10; R, is an

AVI 10 VI alumno7 10 including in the dispersion a fluorochemical emulsifier an aqueous liquid dispersion, comprising the step of This invention also provides a method of stabilizing

$$\begin{cases} H \{ + c - c + c + c - c + 1 \} \\ 0 & R_1 & R_2 \\ 0 & R_2 & R_3 & R_3 \\ 0 & R_2 & R_3 & R_3 \\ 0 & R_2 & R_3 & R_3 \\ 0 & R_3 & R_3 & R_3 \\ 0 & R_2 & R_3 & R_3 \\ 0 & R_3 & R_3 \\ 0 & R_3 & R_3 \\ 0 & R_3 & R_3 & R_3 \\ 0 & R_3 & R_3 \\$$

and wherein W represents the hydrophilic portion of Formuls III are bonds to W in Formulas IV and IVA), obvious proviso that all bonds defined as bonds to Z in defined above in connection with Formula III (with the wherein R1, R2, R3, Q, R, E, X, a, b, and m are as

2) a cationic surfactant; 1) an anionic surfactant;

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3) a non-ionic surfactant; or

4) an amphotene surfactant,

of the invention. emulsifiers find particular use in the aqueous dispersions in an amount effective to stabilize the dispersion. These

sulfur analogs thereof. lene, guanidinylene, oxazolidinonylene, acylimino, and are ureylene, urethanylene, allophanatylene, biuretymer, or the like. Preferred among such linking groups 60 reocyanate or with an isocyanate dimer, trimer, oligophile, such as an alcohol, an amine, or a thiol, with an linking group can result from the reaction of a nucleonate-derived linking group. The isocyanate-derived the organic moiety are linked together by the isocya- 55 substrate. The fluorochemical oligomeric portion and property when the compound is applied to a fibrous soft hand, stain release, water repellency, or a durable group, an organic moiety, and a group that can impart portion, a non-polymeric isocyanate-derived linking 50 four principal portions: a fluorochemical oligomeric comprises a fluorinated compound that generally has las I-III, a fluorochemical composition of the invention As described above and further illustrated in Formu-

one another and preferably contains from about 5 perity of Kygroups (e.g., from 2 to about 50) proximal to herein as N. A fluorinated compound contains a pluralmeric portion is the fluoroaliphatic group, designated 65 A salient component of the fluorochemical oligoimpregnated edgings obtained in this way were coated with an aqueous acid curing varnish (see above) and dried at a temperature of 160.degree. C. for 60 seconds. The varnish addon was about 16 g/m.sup.2. With regard to their performance properties, each of the edgings obtained met the requirements.

[0041] To determine the formaldehyde emissions, the edging samples were conditioned for 3 days under standard climatic conditions (23.degree, C., 50% relative atmospheric humidity) prior to each measurement. To determine the formaldehyde emissions under tropical climatic conditions, the edgings were stored for 1 week at 35.degree, C. and 90% relative atmospheric humidity, after which they were reconditioned for 3 days under standard climatic conditions, prior to the actual measurement. The formaldehyde emissions were measured in accordance with EN 717-2. Table 3 lists the resulting formaldehyde emissions in mg/(h.multidot.m.sup.2) as averages of the 1- to 4-hour values:

3 TABLE 3 Auxiliary as 100% Directly After substance PEG 400 after prep. 1 week Inventive 20 g BC 20 g 0.97 2.61 Example 6 Inventive 20 g BD 20 g 1.28 2.63 Example 7 Comp. Ex. 6 -- 40 g 1.52 3.49 Comp. Ex. 7 20 g polypropylene 20 g 1.82 2.93 glycol (M.sub.n < 400 g/mol)

Inventive Examples 8 and 9 and Comparative Examples 8 and 9

[0042] 1.6 g of 45% acrylic dispersion and 1.0 g of p-toluenesulfonic acid were added to 100 g of the above-described MF resin, along with the auxiliaries indicated in Table 4 for the individual application examples. Following dilution to a liquor concentration of 75%, these liquors were used to impregnate papers (typical edgebanding cardboard; 200 g/m.sup.2) and the impregnated edgings were dried at 180.degree. C. for 90 seconds. The final weight was about 335 g/m.sup.2. With regard to their performance properties, the uncoated edgings obtained met the requirements.

[0043] To determine the formaldehyde emissions, the edging samples were conditioned for 3 days under standard climatic conditions (23.degree. C., 50% relative atmospheric humidity) prior to each measurement. To determine the formaldehyde emissions under tropical climatic conditions, the edgings were stored for 1 week at 35.degree. C. and 90% relative atmospheric humidity, after which they are reconditioned for 3 days under standard climatic conditions, prior to the actual measurement. The formaldehyde emissions were measured in accordance with EN 717-2. Table 4 lists the resulting formaldehyde emissions as averages of the 1- to 4-hour values. The flexibility of the edgings was assessed at room temperature with the aid of the flexural test. The parameter reported was the band radius at which the edging still just did not fracture. As is evident from Table 4, the formaldehyde emissions can be reduced significantly relative to Comparative Example 8 while retaining a very low band radius. The desired formaldehyde reduction cannot be achieved by adding small amounts of urea and sorbitol. Added at higher levels (Comparative Example 9a), there is a deterioration in the flexibility of the edging (larger band radius).

[0044] The table indicates the formaldehyde emission in mg/(h.multidot.m.sup.2) and the band radius in mm.

4 TABLE 4 Auxiliaxy Directly Band as 100% PEG after After radi- substance 400 Sorbitol Urea prep. 1 wk us Inventive 9 g BC 27 g -- -- 0.64 1.57 5 Example 8 Inventive 9 g BC 27 g 1.1 g 1.1 g 0.49 1.51 5 Example 9 Comp. Ex. 8 -- 36 g -- -- 0.97 2.51 6 Comp. Ex. 9 -- 36 g 1.1 g 1.1 g 0.80 2.22 5 Comp. Ex. -- 36 g 3.8 g -- -- 8 9a

Inventive Examples 10 and 11 and Comparative Examples 10 and 11

[0045] 1.7 g of 45% acrylic dispersion and 1.3 g of p-toluenesulfonic acid were added to 100 g of the above-described MF resin, along with the auxiliaries indicated in Table 5 for the individual application examples. Following dilution to a liquor concentration of 75%, these liquors were used to impregnate papers (typical edgebanding cardboard; 200 g/m.sup.2) and the impregnated edgings were dried at 180.degree. C. for 90 seconds. The final weight was about 335 g/m.sup.2. With regard to their performance properties, the uncoated edgings obtained mut the requirements.

[0046] To determine the formaldehyde emissions, the edging samples were

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TITLE: Bis-(2,2,6,6-tetramethyl-piperidyl) diimides as light stabilizers for synthetic polymers

BSPR:

or Y and R.sub.7 together with the N-atom to which they are attached form a succinimide ring which is unsubstituted or substituted by alkyl having 1 to 12 carbon atoms, a maleimide, dimethylmaleimide, phthalimide, tetrahydrophthalimide or hexahydrophthalimide ring,

BSPR:

Preferred compounds of formula I are those wherein if n is 1 Y and R.sub.7 together with the N-atom to which they are attached form a succinimide, maleimide, dimethylmaleimide or phthalimide ring,

BSPR:

Synthetic resins that can have their resistance to deterioration enhanced with bis-(piperidyl) diimides according to this invention include .alpha.-olefin polymers such as polyethylene, polypropylene, polybutene, poly-3-methylbutene, or mixtures thereof and copolymers with other monomers such as ethylene-vinyl acetate copolymer; ethylene-propylene copolymer; polystyrene; polyvinyl acetate; polyacrylic esters; copolymers from styrene and another monomer (for example, maleic anhydride, butadiene, and acrylonitrile); acrylonitrile-butadiene-styrene copolymer, acrylic acid ester-butadiene-styrene copolymer, methacrylic acid ester-butadiene-styrene copolymer, polymethacrylate esters such as polymethacrylate; polyvinyl alcohol; polyvinyl formal; polyvinyl butyral; linear polyesters, polyamides; polycarbonates; polyacetals; polyurethanes, cellulosic resins; phenol-formaldehyde resins; urea-formaldehyde resins; melamine-formaldehyde resins; epoxy resins; unsaturated polyester resins; silicone resins; halogen-containing resins such as polyvinyl chloride, polyvinylidene chloride, polyvinylidene fluoride, and copolymers thereof, and rubbers such as isoprene rubber, butadiene rubber, epichlorohydrin rubber, chloroprene rubber, and blends of any of the above.

RSPR :

The stabilizer or combination is incorporated in the polymer in suitable mixing equipment, such as a mill or a Banbury mixer. If the polymer has a melt viscosity which is too high for the desired use, the polymer can be worked until its melt viscosity has been reduced to the desired range before addition of the stabilizer. Mixing is continued until the mixture is substantially uniform. The resulting composition is then removed from the mixing equipment and brought to the size and shape desired for marketing or use.

BSPR:

The polymer materials stabilized with the stabilizer of the invention can be any physical forms, including filaments, yarns, films, sheets, molded articles, latex, foams and coatings.

8. A stabilizer composition for synthetic resins comprising:

CLPR:

9. A polyvinyl chloride resin composition having improved resistance to deterioration comprising a polyvinyl chloride resin and bis-(2,2,6,6-tetramethy) piperidyl) diimide in accordance with claim 1.

water repellency, or a durable property. nunctional group that imparts soft hand, stain release, reacted, and if the compound used in step (2) contains a all isocyanato groups of a di- or polyisocyanate are nseq in step (2) contains only one isocyanato group or if composition of the invention results if the compound unreacted isocyanato groups in the product. A final

the invention. guaridine that can be present in a final composition of an amine-functional fluorochemical oligomer to form a CO2 affords a carbodiimide, which can then react with sation of two moles of an organic isocyanate with loss of present in a composition of the invention. Also, condenures, e.g., a buiret, can also be formed and can also be final composition of the invention. Derivatives of this affords a urea as a compound that can be present in a tional fluorochemical oligomer with a monoisocyanate of the invention. The reaction of a monoamine-funcreaction and can also be present in a final composition thane, e.g., an allophanate, can also be formed in such a composition of the invention. Derivatives of this ureisocyanate affords a urethane as a compound in a final that contains only one hydroxyl group with the monofor example, the reaction of a fluorochemical oligomer In a cese where a monoisocyanate is used in step (2),

and sultur anologs thereof can be formed. ureas, allophanates, biurets, guanidines, oxazolidinones, have been reacted. As discussed above, urethanes, final compound of the invention if all isocyanato groups tion if there remain unreacted isocyanato groups, or a step (2) will be an intermediate compound of the inven-In the case of di- and polyisocyanates, the product of

with a silicone compound, or by other conventional olefin, by a platinum catalyzed hydrosilation reaction further elaborated, e.g., by polymerization with another isocyanate in step (2), the olefinic unsaturation can be nate. In a product resulting from the use of such an M-IMI from American Cyanamide), and allylisocyamethylethyl)-3-(1'-methylethenyl)benzene (available as furnarate, methacroyl isocyanate, 1(1'-isocyanato-1'isocyanatocthyl methacrylate, bis(2-isocyanato ethyl)isocyanates containing olefinic unsaturation such as diisocyanates such as isophorone diisocyanate (IPDI); polymethylenepolyphenylisocyanate (PAPI); cyclic triisocyanate; aromatic polyiisocyanates such as phatic triisocyanates such as 1,3,6-hexamethylenethylenediisocyanate, and 1,2-ethylenediisocyanate; ali-2,2,4-trimethyl-1,6-hexamethylenediisocyanate, cyanates such as methylenediisocyanate, 1,6-hexamemethods known to those skilled in the art to provide an 50 yl-3,5,5-trimethylcyclohexylisocyanate; aliphatic diisonate; alicyclic diisocyanates such as 3-isocyanatomethdisocyanate, and 1,8-dinitro-2,7-naphthylene disocya-4-chloro-1,2-naphthylene diisocyanate, 1,3-naphthylene diisocyanatobenzene, 1,2-naphthylene diisocyanate, dipbenyl, dimethyl-4,4'-diisocyanatodiphenyl, 2,2'-dichloro-5,5'benzyl, 3,3'-dimethoxy-4,4'-diisocyanatodiphenyl, 3,3'thane, 4,5'-diphenyldiisocyanate, 4,4'-diisocyanatodi-3,3'-dichloro-4,4'-diisocyanatodiphenylmeylether, 4,4'-disocyanatodiphendiisocyanate, 40 p-xylylene tolunediisocyanate, 2,6-toluene diisocyanate, o, m, and diisocyanate, 77 fluoromethyl)-1,3-benzene 4,4'-methylenediphenylenedüsocyanate, -in)-ib-0,4 octadecylisocyanate; aromatic diisocyanates such as juclude monoisocyanates such as phenylisocyanate and Examples of isocyanates suitable for use in step (2)

> V-601 (ester-functional), and V-501 (acid-functional). (alkylamino-functional), VR-041 kanol-functional),

cent by weight of an initiator can be used, based on the about 0.8 percent, and most preferably about 0.2 percent to about 5 percent, preferably about 0.1 percent, to 10 initiator and other reactants being used. About 0.1 persuitable amount of initiator depends on the particular bly between about 0.05 to about 0.25. Otherwise, a mers is between about 0.01 to about 0.5, more preferasuch that the molar ratio of initiator to olelinic monoinitiator, it is preferred to use the initiator in an amount When a compound is to be functionalized via the

chloroform, FREON TM 113, trichloroethylene, a,a,athylacetamide), halogenated solvents such as methylamides (e.g., N,N-dimethylformamide, N,N-dimeisobutyl ketone), sulfoxides (e.g., dimethyl sulfoxide), hol), ketones (e.g., acetone, methylethyl ketone, methyl 25 butyl acetate), alcohols (e.g., ethanol, isopropyl alcoglyme, disopropyl ether), esters (e.g., ethyl acetate, toluene, xylene), ethers (e.g., diethylether, glyme, ditane, cyclohexane), aromatic solvents (e.g., benzene, aliphatic and alicyclic hydrocarbons (e.g., hexane, hep- 20 reaction mixture. Examples of suitable solvents include percent by weight based on the total weight of the concentration, e.g., from about 5 percent to about 90 reactants can be present in the solvent at any suitable solvent suitable for organic free-radical reactions. The Step (1) of either Scheme can be carried out in any total weight of all other reactants in the reaction.

ators and all solvents, generally suitable temperatures enumerate a particular temperature suitable for all inititicular initiator, and the like. While it is not practical to reagents, the temperature required for the use of a parthe art based on considerations such as the solubility of 35 vents for use can be easily selected by those skilled in free-radical reaction. Particular temperature and solat any temperature suitable for conducting an organic Likewise, step (1) of either Scheme can be carried out trilluorotoluene, and the like, and mixtures thereof.

a hydroxy-functional compound can be oxidized by both hydroxy and chloro groups. As a further example, epichlorohydrin to provide a further compound with droxy-functional compound can be reacted with an ambit of the respective formula. For example, a hy. 45 dimethoxy-4,4'-diisocyanato functional groups, the product still being within the conventional methods to include further or different (Scheme I) or VIII (Scheme II), can be elaborated via The product of step (1), a compound of Formula VII are between about 30° C. and about 200° C.

to provide an amine-functional compound also contain-

acid-functional compound, or reacted with caprolactam

position of the invention results if there remain any tion can be prepared by step (2). An intermediate commer. Intermediate and final compositions of the invenof a single di- or polyfunctional fluorochemical oligopendently reacting with a nucleophilic functional group two or more organic isocyanate molecules each indesented by compounds of Formula IIA, is the result of of a single di- or polyisocyanate. The other class, repreeach independently reacting with an isocyanato group 60 the result of two or more fluorochemical oligomers represented by coupounds of Formulas IA and IIIA, is one of two general classes of compounds. One class, to form the linking group Z. Step (2) diverges to afford VIII (Scheme II) is reacted with an organic isocyanate 55 In step (2) a compound of Formula VII (Scheme I) or ing an ester group.

CLPR

16. A polyvinyl chloride resin composition in accordance with claim 9 in which the polyvinyl chloride resin is polyvinyl chloride homopolymer.

CLPR:

11. A polyvinyl chloride resin composition in accordance with claim 9 in which the polyvinyl chloride resin is a copolymer of vinyl chloride and vinyl acetate.

CLPR:

12. An olefin polymer <u>composition</u> having improved resistance to deterioration comprising an olefin polymer selected from the group consisting of polymers of alpha-olefins having from two to six carbon atoms and polystyrene, and bis-(2,2,6,6-tetramethyl piperidyl)diimide in accordance with claim 1.

CLPR:

13. An olefin polymer <u>composition</u> in accordance with claim 12 wherein the polyolefin is polypropylene.

CLPR:

14. An olefin polymer <u>composition</u> in accordance with claim 12 wherein the polyolefin is polyethylene.

CLPR:

15. An olefin polymer composition in accordance with claim 12 wherein the polyolefin is ethylene-propylene copolymer.

CLPR -

16. A polyurethane resin <u>composition</u> having improved resistance to deterioration comprising a polyurethane resin and bis-(2,2,6,6-tetramethyl piperidyl)diimide in accordance with claim 1.

afford the blocked isocyanato group. blocking agent, c.g., an oxime such as acetone oxime, to

isocyanate-derived group formed in step (3); Z is the chanate used in step (3) bonded thereto through the in step (2) with the organic nucleus of the di- or polyisoorganic moiety R includes the blocked oligomer formed to form a linking group Z. In such a compound, the with a fluorochemical oligomer of Formula VII or VIII reacting the remaining isocyanate groups of the product also containing blocked isocyantato groups; and (4) ford an isocyanato-containing intermediate compound oligomer with an organic di- or polyisocyanate to atan isocyanate; (3) reacting a deficiency of the functional groups and a functional group capable of reacting with blocked oligomer containing blocked isocyanato functional end-capping agent to provide a functional blocked monomer in the presence of an appropriate late to afford a blocked monomer; (2) oligomerizing the functional monomer such as isocyanatoethyl methacry-(1) blocking the isocyanato group of an isocyanateganic moiety R contains a blocked isocyanato involves paring a compound of this invention wherein the or-Another method (not shown in the Schemes) of pre-

Such can be introduced in step (3) by, for example, Organic moiety R can contain other types of groups. mula VII or VIII above. the remainder of the compound is as defined for Forisocyanate-derived linking group formed in step (4); and

triecthylenetetramine, ethylenetriamine, dihexylamine, ethylenediamine, 1,6-hexanediamine, didi, or polyamines such as butylamine, dibutylamine, 1,4-butanediol, 1,6-hexanediol, 1,10-decanediol; mono, ene glycol, glycerol, 1,2,4-butanetriol, pentaerythritol, cthanol, butanol, 2-ethylhexanol, stearylalcohol, ethylproic acid; mono, di, or polyalcohols such as methanol, sulfonic acid, taurine; amino acids such as 6-aminocasuitable temperatures are between about room tempera- 60 acids such as 4-(2-hydroxyethyl)-1-piperazineethane ethyldiethanolamine, aminosultonic cthanolamine, tricthanolamine, N,N-dimethylaminoethanol, methyldisciq' b-n'ydroxybenzoic scid; sminoslcobols such as taric acid, dimethylolacetic acid, dimethylolpropionic scid, glyoxal, 12-hydroxystearic acid, citric acid, tarable as PECHTM, 3M); hydroxyacids such as malic chlorohydrin) mono-, di-, or triols (such as those avail-Dow Corning 531 and 536 (polyamines), poly(cpi-Corning Q4-3557 (polyol), Dow Corning 8026 (polyol), as Dow Corning Q4-3667 (diol), Dow Corning X-2propanediol; mono, di, or polyfunctional siloxanes such propanol, 2,3-dibromopropanol, and 1-chloro-2,3-1,3-dichloro-2-chloroethanol, methylaziridine, incorporated herein by reference; functionalized alcoal.), and 4,606,737 (Stern) the disclosures of which are (Husted), 2,691,043 (Husted), 3,398,182 (Guenther et those disclosed, for example, in U.S. Pat. Nos. 2,666,797 acid; fluorochemical alcohols, amines, and thiols such as adipic acid, benzoic acid, phthalic acid, terephthalic sorbic acid, linoleic acid, oxalic acid, succinic acid, such as lauric acid, palmitic acid, stearic acid, oleic acid, ples of reagents suitable for use in step (3) include acids resctive group will generally remain unreacted. Examof reactivity with isocyanates than the others. The less of the functional groups has a substantially lesser degree functional groups is used in such a reaction, at least one nate. When a reagent containing two or more reactive with a reagent containing a group reactive to an isocyareacting an isocyanate of Formulas IA, IIA, or IIIA

> as DESMONDUR TM R) are suitable. tri-(4-isocyanatophenyl)-methane (available from Bayer SMONDUR TM TT. Also, other trisocyanates such as disocyanates such as that available from Bayer as DE-Germany, as IPDI-1890, and azeredinedione-containing tri-isocyanates such as that available from Huls AG, DESMONDUR TM N-100, isocyanurate-containing tri-isocyanates such as that available from Mobay as isocyanate-derived moieties such as biuret-containing Also useful are isocyanates containing internal

the three isophoronyl substituents thereon; n will be 3; derivative isocyanurate nucleus of the IPDI-1890 and Formula I or III can be considered to be the isocyanate- 15 Formula VII, the organic moiety R in the product of with a hydroxy-functional oligomeric intermediate of nate in Step (2), and all isocyanato groups are reacted When, for example, IPDI-1890 is used as the isocya-

Other suitable types of isocyanates for use in step (2) and each Z will be a urethanylene group.

comprises a fluoroaliphatic moiety. afford a composition wherein the organic moiety R siloxyl moiety. Also, fluorochemical isocyanates such as Fi7C85O3—(CeH4)—NCO can be used, which will 25 composition wherein the organic moiety R comprises a taining amines with phosgene and which will afford a nates, which can be prepared by reacting siloxane-coninclude siloxane-containing mono, di, or polyisocya- 20

weight based on the total weight of the reactants. cent, preferably about 0.1 percent to about 5 percent, by trations are from about 0.001 percent to about 10 per- 50 8024 (dimercaptan), Dow Coming 1248 (polyol), Dow trations. Generally, however, suitable catalyst concenit is not practical to recite particular preferred concenpresent will depend on the particular reaction, and thus out in the absence of catalyst. The amount of catalyst such as triethylamine. Urea formation can be carried 45 hols and amines such as glycidol, aziridine, 2while allophanate formation can be catalysed by amines octosic are preferred in the preparation of urethanes, reaction. For example, tin catalysts such as stannous isocyanate derivative that is the intended product of the aromatic nature of the isocyanate) and on the particular 40 the fluorochemical oligomer and the aromatic or non-(e.g., the nature of the nucleophilic functional groups in art. The preferred catalyst depends on the reactants nous chloride; and others known to those skilled in the stannous oleate, tin dibutyldi-(2-ethyl hexanoate), stan- 35 salts such as dibutyltin dilaureate, stannous octoate, methyldicthanolamine; N,N-dialkylalkanolamines; tin line, M,N',N',-tetramethyl ethylene diamine, Mtriethylamine, triethylenediamine, N-methylmorpholyst. Suitable catalysts include tertiary amines such as 30 Preferably the reaction is run in the presence of a cata-Step (2) is carried out under conventional conditions.

Oct mode and about 200° C. ular temperatures suitable for all situations, generally being used. While it is not practical to enumerate particbased on the particular reagents, solvents, and catalysts tures will be essily determined by those skilled in the art isobutyl ketone, and the like. Suitable reaction tempera- 55 in a polar solvent such as ethyl acetate, acetone, methyl Step (2) is preferably carried out under dry conditions

IA, IIA, or IIIA can be reacted with an isocyanate prepare such compounds, an intermediate of Formula I, II, or III can contain blocked isocyanato group(s). To scribed above, the group R of a compound of Formula 65 pound of Formula I, II, or III respectively. As de-Formula IA, IIA, or IIIA is reacted to form a com-In step (3) of Schemes I and II, an intermediate of